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STUDIES ON THE STORAGE OF WHEATEN FLOUR: I. THE INFLUENCE OF STORAGE ON THE CHEMICAL COMPOSITION AND BAKING QUALITY OF FLOUR[1]

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It is well known that wheat flours improve in baking quality during storage. This improvement continues for some time until it reaches a maximum, after which progressive deterioration sets in, the flour becoming eventually unfit for bread-making. Flours, even when made from good sound wheat, vary widely in their response to storage. In one instance samples of well-milled straight run No. 1 N. Manitoba and all-English flours, the latter made from a mixture of ordinary red English varieties (not Yeoman), were kept in the laboratories for 2 years in galvanised iron bins. At the end of this time the Manitoba flour made a first-class loaf. The English flour had become thoroughly broken down, the resulting loaf being small and most unappetising; it was in fact a loaf only in name. Photographs of these two loaves are shown as Figure 1.

One of the most interesting studies of the effect of storage under good conditions on baking quality was carried out by Saunders (see Saunders *et al.*, 1921) in Canada. He stored both wheats and flours for periods up to 14 years in some cases. The results may be summarised in his own words:

"The residues from a number of samples of flour which had been baked were kept for a year longer, in a room which was warmed in winter. On repeating the baking tests the following winter, very marked improvement was noted in nearly all cases. Indeed, in the very first instance the change in the flour by twelve months' storage was so radical that it was supposed there must have been some

[1] Condensed from Confidential Report No. 18 of the Research Association of British Four-Millers issued in October, 1930. Released for publication by the Council of the Research Association, 1935.

error. However, further work showed the undoubted correctness of the early observations."

Several extensive series of investigations were carried out on many varieties of wheat and flour. Saunders concluded:

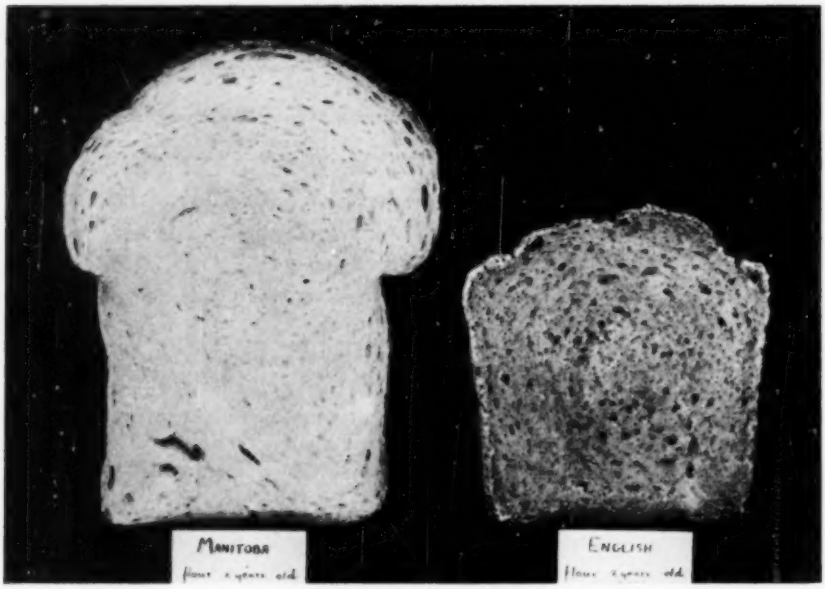

Fig. 1. Cross-sections of loaves made from Manitoba and English flours after 2 years' storage.

"Most of these varieties of wheat improved very considerably in baking qualities by long storage, either as wheat or as flour. The highest baking strength was attained when the wheat was stored about three or four years. When the material was stored as flour the changes were more rapid and the highest baking strength was reached in two or three years. It appears that by storing the material as flour a higher baking strength can be obtained than is possible when the material is stored as wheat. Ultimately, deterioration sets in, rather earlier with the flour than with the wheat; but it is evident that storage under good conditions is safe for at least ten years.[2] The amount of water absorbed by the very old samples of flour is phenomenal, as is also their extremely white colour. The dough made from these (very old) samples was quite 'short' and lacked the stickiness necessary to reach a maximum loaf volume. Evidently important changes in the gluten had taken place during the long period of storage."

Some of the most typical of Saunders' results are summarised in Table I, which should be studied in connection with the above summary.

Saunders' final conclusions were as follows:

"While the evidence . . . is not all in perfect harmony and is not conclusive on every point, it clearly establishes some facts which are quite contrary to prevalent ideas.

"Storage of new wheat, or of new flour, for a few months after harvest gives an increase in water absorbing power and, in the case of flour at any rate, a decided improvement in colour, a natural bleaching. There is, however, little

[2] This conclusion only applies to the Canadian varieties studied by Saunders. It is not true for English varieties of wheat.

TABLE I

EFFECT OF STORAGE ON BAKING QUALITY OF FLOURS (SUMMARISED FROM SAUNDERS)

When baked	Water absorption	Loaf volume	Crumb colour	Baking strength
	%	*Cc.*	*Score*	*Score*
Flour from Huron Wheat:				
Sept., 1907	58.3	425	88	86
Jan., 1908	61.3	423	90	91
Jan., 1909	62.5	465	94	99
Jan., 1910	62.8	509	102	107
Jan., 1911	64.3	512	102	107
Mar., 1912	66.2	463	102	98
April, 1919	68.8	392	102	91
Jan., 1921	74.0	377	102	87
Flour from Red Fife Wheat:				
Sept., 1907	58.1	522	100	98
Jan., 1908	62.4	482	100	98
Jan., 1909	62.8	528	104	107
Jan., 1910	63.4	532	104	109
Jan., 1911	63.9	511	105	106
Mar., 1912	66.0	476	104	102
April, 1919	70.3	461	104	102
Jan., 1921	75.1	417	102	95
Flour from Yellow Cross Wheat:				
Sept., 1907	56.9	394	90	74
Jan., 1908	58.4	381	92	77
Jan., 1909	59.3	475	100	101
Jan., 1910	60.9	535	104	109
Jan., 1911	62.3	537	104	111
Mar., 1913	63.0	495	103	103
April, 1919	68.0	473	103	100
Jan., 1921	72.0	443	103	100

All three wheats were ground on September 16th and 17th, 1907.

or no change in volume, texture, and shape of loaf. The baking strength of the flour remains practically the same.

"The improvement of wheat or flour by long storage is quite marked in practically all cases and in many instances amounts to nothing less than a complete transformation after a couple of years.

"Deterioration of stored wheat or flour does not begin until after a period of several years. The storage conditions must, of course, be good, for doubtless in a damp, unventilated room deterioration might set in very early."

It should be noted that the deterioration referred to is purely relative, *i.e.*, relative to the condition of the flour after maximum improvement had been attained. With very few exceptions the wheats and flours studied by Saunders were of better baking quality, or, at least, as good, at the end of the storage periods than they were initially. One striking exception was a high-grade commercial patent flour milled in mid-winter, 1910. As shown by the following figures the baking quality of the flour fell below its initial value after nearly ten years of storage:

When baked	Water absorption of dough	Loaf volume	Baking strength
	%	*Cc.*	
January, 1911	65.0	500	104
April, 1919	67.6	502	107
January, 1921	75.0	455	97

These investigations of Saunders have been described at some length as they are among the best and most conclusive investigations carried out in this field by North American workers. Other, more limited, investigations have either supported or have been at least consistent with those of Saunders. Among these may be mentioned those of Stockham (1907), Harcourt (1909), Fitz (1910), Shutt (1911), Swanson, Willard, and Fitz (1915), and Whitcomb, Day, and Blish (1921).

The outstanding fact established by these studies is the enormous periods of time for which flours made from hard Canadian and American wheats may be stored before the flours become unfit for bread-making. This is not in accord with the experience of English millers, who are only too well aware of the difficulties of keeping flour for any prolonged period even under really good storage conditions. No commercial flour made in England from a mixed grist could be stored under normal conditions for a period even remotely comparable to 10 years, while an all-English flour would become unfit for bread-making in comparatively few months.

Marion (1909), using presumably French wheats, showed that although the flour retained its baking qualities for 3 months, it was not suitable for baking after 6 or 7 months. The amount of crude gluten obtainable by washing out showed only slight fluctuations up to the 19th month of storage after which it became very difficult to recover gluten as a coherent mass.

It has been shown by the writers that No. 1 N. Manitoba flour may be kept up to 5 years without any deterioration taking place, while, as mentioned above, an all-English flour had become thoroughly broken down and completely unfit for bread-making in less than 2 years.

These wide differences in response to storage between hard, strong North American wheats and soft, weak western European wheats are striking, and are of peculiar interest to English millers on account of the immense variety of wheats used by the English milling industry. The great majority of English-milled flours are made from wheat mixtures containing not only different wheats, but different grades of nominally the same wheats. Moreover, nothing is known concerning the influence of one constituent of the grist on the others in so far as response to storage is concerned.

The influence of storage on flour quality is of importance from another point of view. The use of the so-called chemical improvers and chemical improvement processes originated in the desire of millers to expedite or to imitate the natural improvement brought about in flour by more or less lengthy periods of storage. Any information that could be obtained concerning the changes taking place in flour as a result of storage might be of value in elucidating the action of flour improvers.

Numerous studies of limited scope have been made by various workers of the changes in certain chemical characters of flour incident to storage among whom may be mentioned Snyder (1904), Leavitt and LeClerc (1909), Sanderson (1913), Sharp (1924), Bailey and Johnson (1924), and Greaves and Hirst (1925).

In this paper observations are described which were made on stored flours over a period of 18 months.[3]

Experimental

Types of Flours Used and Preparation of Samples

In this investigation two flours were studied, a No. 3 N. Manitoba and an all-English, non-Yeoman, type. Each flour was stored in the form of two divides, a top 60% patent and a 40% low-grade, and each divide was stored in tins at three different moisture contents, approximately 12%, 16%, and 18% respectively—twelve samples in all. The remainder of the four flours was kept in the original bags in the flour store. Analytical determinations were carried out periodically on all sixteen samples and baking tests were carried out on the four bagged samples.

The No. 3 N. Manitoba wheat was a good average specimen of the grade. On arrival at the mill it contained 12.84% moisture. The first break feed contained approximately 16¼% water. The wheat was milled on January 3rd, 1929, in cold weather when the inside temperature of the mill was below normal—say about 50° F. on the mill floors. The extraction calculated as percentage of total products was about 72%. The 60% patent flour contained 15.59% moisture and the 40% low-grade 15.50% moisture at time of milling.

The English wheats were drawn from commercial bulks representing an average of several lots. The moisture contents of these wheats on arrival at the mill ranged from 18.11% to 22.65%, the average natural moisture content being about 19.5%. The wheat milled was washed with a short immersion and then dried so that the moisture content at the first break was 16.25%. The wheat was milled on January 8th in cold weather, the air temperature being about the same as for the Manitoba. The patent flour contained 16.12% and the low-grade 15.82% moisture at time of milling.

Each flour was thoroughly mixed by hand at the laboratories.

To obtain the dry samples of moisture content of approximately 12%, the samples were spread out in thin layers in the laboratory for several days, moisture tests being made at intervals, after thorough mix-

[3] A number of papers have appeared since 1930, the date of the work described herein. They need not be enumerated here as they deal with other aspects of the problem than those discussed in the present communication.

ing of each sample, until the desired moisture contents had been reached.

Other samples were spread in thin layers on trays in a Carrier processing cabinet and air, which had been passed through water sprays so as to materially raise its humidity, was circulated over the layers at the ordinary laboratory temperature until the desired moisture content—approximately 18%—had been reached.

Type of Storage Containers Used

Each lot of flour was then thoroughly mixed and stored in cubical sealed tins of about 15-pound capacity. The tins were placed in a large zinc lined wooden case with considerable packing material between the tins and the walls of the case so as to reduce to some extent the fluctuations in temperature. Maximum and minimum thermometers were enclosed in the case. During the whole period of storage the temperature inside the case varied between 43° F. and 77° F.

Nature of Tests Made on Flours

The preparation of the samples took a considerable time and the first series of analyses was carried out on February 9th, 4 to 5 weeks after milling. The analytical results are given in Table II. After this the

TABLE II

ANALYSES OF THE FOUR FLOURS BEFORE COMMENCEMENT OF STORAGE

Flours were four to five weeks old. All results except moisture are calculated to 16% moisture basis.

	Manitoba		English	
	Patent	Low grade	Patent	Low grade
Moisture, %	14.1	14.3	16.0	15.9
Ash, %	0.383	0.780	0.403	0.492
Total nitrogen (N), %	1.90	2.20	1.43	1.43
Total protein (N × 5.7), %	10.83	12.54	8.16	8.16
Dry gluten (by washing out), %	12.1	14.1	8.8	8.6
Soluble nitrogen, %	0.223	0.275	0.214	0.205
Nitrogen soluble in 5% potassium sulphate solution, %	0.265	0.413	0.240	0.279
Total phosphorus (P_2O_5), %	0.241	0.448	0.198	0.286
Soluble phosphorus, %	0.044	0.173	0.034	0.091
Soluble extract, %	4.90	6.35	3.56	3.97
Maltose figure, %	1.96	2.05	0.95	1.07
Total acidity, %	0.26	0.37	0.30	0.33
pH	6.11	6.41	6.10	6.27
Buffer value	0.73	0.36	0.90	0.64

tinned and bagged samples were analysed and baked at first at monthly, and later at bi-monthly intervals. In addition to the baking tests the following analytical determinations were carried out on the sixteen samples: moisture, soluble extract, soluble phosphorus (P_2O_5), soluble nitrogen, nitrogen soluble in 5% potassium sulphate solution, maltose

figure, dried gluten (by washing out), total or titratable acidity, hydrogen ion concentration and buffer value (of 10% aqueous suspensions), amino-acid content and proteoclastic activity.

Results of Laboratory Tests

The full results are given for reference in Tables III to VIII. The results show some irregularities, due possibly to irregular mixing and sampling and to irregular ageing in different parts of the samples, but the general trend is clearly brought out by the tables.

Effect of Type of Storage Container

The bagged samples dried considerably during the first month, after which their moisture contents fluctuated between 12.5% and 14%. The tinned samples also showed some fluctuations in moisture contents. Throughout the first 6 months the bagged samples averaged 13.2% moisture, and the tinned dry, normal and moist samples 11.7%, 16.2% and 18.3% respectively. As will be seen from the tables, the bagged and tinned dry samples showed closely similar changes in chemical composition during 18 months storage, and the changes were by no means marked. On the whole, the tinned dry samples showed smaller changes than the corresponding bagged (and slightly moister) samples. The Manitoba dry and bagged samples showed greater changes than the English, and in each case the low-grade showed greater changes than the patent flours.

Flour Acidity

These figures also bring out strikingly the great effect of moisture content on the changes in acidity. The normal samples with 16% moisture show much more rapid changes in acidity than the drier samples with 13% and 12% moisture, and these changes became particularly rapid after 5 to 6 months with the two English samples and after 2 months with the Manitoba flours. With the Manitoba flours a maximum acidity was reached at 7 months in the case of the patent flours and at 4 months with the low-grade, after which equally rapid decreases occurred until after 11 months storage the original initial acidities were regained. The changes in the English normal flours took place more slowly than in the Manitoba, but the rapid slowing up of the changes in the English flours after 10 months or so suggests that with these flours, too, after sufficiently prolonged storage maximum acidities would be reached and followed by subsequent decreases. Similar changes in acidity occurred, but more rapidly, with the moist samples of moisture content 18%. With these samples maxima, followed by minima, occurred

TABLE III

EFFECTS OF STORAGE AT DIFFERENT MOISTURE CONTENTS ON CHEMICAL COMPOSITION OF FLOUR. ENGLISH PATENT FLOUR
(Results Calculated to 16% Moisture Basis)

Period of storage		Moisture when sampled	Soluble extract	Soluble P_2O_5	Soluble nitrogen	Potassium sulphate soluble N	Maltose	Titratable acidity	pH	Buffer value
Weeks		%	%	%	%	%	%	%		
0		16.0	3.56	0.034	0.214	0.240	0.95	0.30	6.10	0.90
6	Tins Dry	11.99	3.21	.033	.222	.225	.93	.20	6.03	0.84
	Tins Normal	15.93	3.56	.035	.224	.222	.93	.23	6.03	0.87
	Tins Moist	17.74	3.86	.039	.267	.222	1.05	.22	5.80	0.77
	Bag	13.50	3.46	.035	.220	.234	.89	.24	6.00	0.83
10	Tins Dry	12.00	3.52	.033	.230	.233	.86	.23	6.02	0.81
	Tins Normal	15.66	3.50	.034	.235	.233	.85	.26	6.05	0.86
	Tins Moist	17.70	5.98	.042	.616	.220	1.23	.37	5.04	0.47
	Bag	13.03	3.51	.035	.232	.226	.97	.23	6.04	0.83
14	Tins Dry	11.94	3.55	.033	.219	.232	.79	.28	6.03	0.82
	Tins Normal	15.84	3.54	.033	.224	.210	.83	.31	6.01	0.80
	Tins Moist	18.15	7.14	.043	.767	.216	1.13	.67	4.64	0.30
	Bag	13.60	3.29	.034	.207	.232	.83	.24	6.01	0.79
18	Tins Dry	11.62	3.57	.034	.212	.234	1.14	.47	6.02	0.86
	Tins Normal	15.67	3.69	.036	.233	.207	1.10	.41	5.95	0.82
	Tins Moist	18.00	5.20	.039	.517	.208	1.23	.48	5.27	0.57
	Bag	13.78	3.76	.031	.220	.240	1.36	.34	5.92	0.79
22	Tins Dry	11.64	3.53	.034	.208	.221	1.33	.37	6.01	0.84
	Tins Normal	15.21	3.72	.035	.235	.217	1.19	.42	5.82	0.73
	Tins Moist	17.95	3.69	.034	.223	.209	1.02	.34	5.81	0.81
	Bag	13.91	3.47	.034	.212	.231	1.07	.34	5.79	0.68

TABLE III—*Continued*

Period of storage			Moisture when sampled	Soluble extract	Soluble P_2O_5	Soluble nitrogen	Potassium sulphate soluble N	Maltose	Titratable acidity	pH	Buffer value
Weeks 0			% 16.0	% 3.56	% 0.034	% 0.214	% 0.240	% 0.95	% 0.30	6.10	0.90
26	Tins	Dry	11.25	4.16	.057	.237	.223	.95	.31	5.99	0.78
		Normal	15.36	5.20	.052	.397	.210	1.09	.39	5.47	0.61
		Moist [1]	18.03	3.63	.049	.187	.208	.82	.25	6.18	0.98
	Bag		13.50	3.28	.033	.208	.206	.78	.32	5.92	—
30	Tins	Dry	11.42	—	—	—	—	—	—	5.95	0.84
		Normal	15.01	6.17	.042	.606	—	1.03	.53	5.20	0.51
		Moist [2]	17.60	—	—	—	—	—	—	6.32	0.96
	Bag		13.73	3.68	.039	.229	—	.78	.43	5.91	0.73
34½	Tins	Dry	—	—	—	—	—	—	—	—	—
		Normal	15.94	7.49	.041	.832	.196	1.2	.77	4.49	0.32
		Moist	—	—	—	—	—	—	—	—	—
	Bag		13.93	3.55	.034	.234	.220	.98	.34	5.83	0.83
39	Tins	Dry	11.52	—	—	—	—	—	—	—	—
		Normal	15.32	—	—	—	—	—	—	4.35	0.29
		Moist [3]	18.06	—	—	—	—	—	—	—	—
	Bag		14.08	—	—	—	—	—	—	6.01	0.99
49½	Tins	Dry [4]	12.17	3.60	.038	.215	.214	.88	.34	—	—
		Normal [4]	15.77	9.21	.051	1.01	.182	1.13	1.01	—	—
		Moist [3]	18.33	2.92	.034	.175	.178	.59	.20	—	—
	Bag [4]		14.14	3.51	.035	.229	.224	.85	.39	5.88	0.78
60	Bag only		14.16	3.64	.035	.253	.219	.83	.44	5.87	0.82
66	Bag only		14.03	3.66	.031	.238	.196	.78	.37	5.81	0.76
78	Tins—Normal only		15.76	7.46	.045	.806	—	1.13	1.09	4.11	.21
	Bag		14.28	3.62	.035	.232	—	.89	.41	5.91	.85

[1] Slight smell of moulds. [2] Strong smell of moulds. [3] Very strong smell of moulds. [4] No marked smell.

TABLE IV

EFFECTS OF STORAGE AT DIFFERENT MOISTURE CONTENTS ON CHEMICAL COMPOSITION OF FLOUR. ENGLISH LOW GRADE
(*Results Calculated to 16% Moisture Basis*)

Period of storage			Moisture when sampled	Soluble extract	Soluble P_2O_5	Soluble nitrogen	Potassium sulphate soluble N	Maltose	Titratable acidity	pH	Buffer value
Weeks			%	%	%	%	%	%	%		
0			15.9	3.97	0.091	0.205	0.279	1.07	0.33	6.27	0.64
6	Tins	Dry	11.77	4.11	.083	.218	.259	1.05	.23	6.32	0.63
		Normal	15.66	3.92	.086	.219	.277	.92	.25	6.28	0.59
		Moist	18.52	4.36	.086	.239	.261	1.34	.62	5.50	0.60
	Bag		13.52	4.23	.079	.220	.271	.96	.32	6.31	0.63
10	Tins	Dry	11.57	3.84	.078	.221	.272	1.14	.28	6.24	0.61
		Normal	15.66	4.01	.088	.217	.258	1.00	.34	6.26	0.63
		Moist	18.62	4.54	.087	.242	.256	1.38	.58	5.40	0.58
	Bag		12.63	4.07	.076	.217	.277	1.06	.27	6.31	0.63
14	Tins	Dry	11.69	3.93	.070	.201	.270	.95	.32	6.28	0.61
		Normal	15.79	3.96	.067	.216	.278	1.0	.37	6.24	0.59
		Moist	18.80	4.79	.070	.261	.248	1.35	.63	5.35	0.49
	Bag		13.58	3.98	.065	.204	.277	.97	.33	6.29	0.59
18	Tins	Dry	11.63	3.92	.075	.200	.264	1.35	.41	6.28	0.67
		Normal	15.81	4.06	.066	.208	.267	1.30	.51	6.14	0.68
		Moist	18.92	4.02	.062	.226	.255	1.45	.46	5.53	0.55
	Bag		14.06	4.08	.066	.200	.268	1.08	.38	6.25	0.61
22	Tins	Dry	11.49	3.94	.067	.199	.258	1.04	.47	6.28	0.67
		Normal	15.44	4.35	.070	.224	.243	1.69	.57	5.67	0.59
		Moist	18.80	3.55	.057	.186	.231	1.45	.31	6.02	0.61
	Bag		13.72	4.04	.072	.205	.271	1.36	.41	6.23	0.65

TABLE IV—*Continued*

Period of storage			Moisture when sampled	Soluble extract	Soluble P_2O_5	Soluble nitrogen	Potassium sulphate soluble N	Maltose	Titratable acidity	pH	Buffer value
Weeks			%	%	%	%	%	%	%		
0			15.9	3.97	0.091	0.205	0.279	1.07	0.33	6.27	0.64
26	Tins	Dry	11.04	4.51	.089	.212	.254	1.04	.23	6.26	0.70
		Normal	15.33	6.22	.081	.376	.238	1.29	.72	4.87	0.32
		Moist [1]	18.96	3.35	.053	.182	.229	.93	.24	6.42	0.56
	Bag		13.11	3.85	.063	.194	.255	1.06	.39	6.22	0.70
30	Tins	Dry	11.17	—	—	—	—	—	—	6.21	0.68
		Normal	14.98	6.06	.058	.510	—	1.38	1.18	4.74	0.46
		Moist [2]	17.26	—	—	—	—	—	—	6.52	0.73
	Bag		14.55	4.17	.077	.205	—	1.08	.37	6.17	0.67
34½	Tins	Dry	—	—	—	—	—	—	—	—	—
		Normal	15.94	7.26	.061	.691	.182	1.4	1.36	4.03	0.13
		Moist	—	—	—	—	—	—	—	—	—
	Bag		14.23	4.01	.068	.206	.238	1.08	.47	6.12	0.74
39	Tins	Dry	11.36	—	—	—	—	—	—	—	—
		Normal	15.38	—	—	—	—	—	—	3.94	0.14
		Moist	—	—	—	—	—	—	—	—	—
	Bag		13.07	—	—	—	—	—	—	6.11	0.69
49½	Tins	Dry [3]	12.85	4.03	.078	.206	.247	1.09	.48	—	—
		Normal [3]	16.07	8.00	.078	.699	.196	1.53	1.52	—	—
		Moist [4]	23.00	6.20	.069	.552	.444	1.53	.41	—	—
	Bag [3]		13.78	3.90	.065	.190	.266	1.05	.51	6.09	0.58
60	Bag only		14.03	4.00	.071	.169	.260	1.08	.53	6.12	0.69
66	Bag only		13.99	3.97	.065	.197	.232	.98	.53	6.09	0.67
78	Tins—Normal only		15.56	4.93	.059	.314	—	1.59	1.00	4.87	.41
	Bag		13.93	4.00	.063	.193	—	1.17	.60	6.12	.73

[1] Marked smell of moulds. [2] Strong smell of moulds. [3] No marked smell. [4] Strong mouldy smell (ammoniacal).

TABLE V

EFFECTS OF STORAGE AT DIFFERENT MOISTURE CONTENTS ON CHEMICAL COMPOSITION OF FLOUR. MANITOBA PATENT FLOUR
(Results Calculated to 16% Moisture Basis)

Period of storage			Moisture when sampled	Soluble extract	Soluble P_2O_5	Soluble nitrogen	Potassium sulphate soluble N	Maltose	Titratable acidity	pH	Buffer value
Weeks			%	%	%	%	%	%	%		
0			14.1	4.90	0.044	0.223	0.265	1.96	0.26	6.11	0.73
6	Tins	Dry	12.14	5.14	.042	.239	.277	2.01	.20	6.09	0.74
		Normal	16.34	5.00	.044	.239	.266	1.50	.21	6.11	0.77
		Moist	18.01	4.95	.045	.268	.272	1.54	.49	5.90	0.69
	Bag		12.89	4.95	.043	.235	.293	1.93	.27	6.09	0.75
10	Tins	Dry	11.96	5.04	.040	.230	.262	1.91	.29	6.06	0.71
		Normal	16.20	4.91	.045	.244	.287	1.70	.20	6.09	0.74
		Moist	18.12	6.72	.043	.498	.235	1.85	.58	5.29	0.51
	Bag		12.35	4.81	.043	.232	.259	1.92	.29	6.13	0.75
14	Tins	Dry	12.44	5.01	.040	.234	.270	1.90	.33	6.08	0.72
		Normal	16.18	5.25	.047	.279	.280	1.60	.41	5.82	0.73
		Moist	18.24	7.31	.040	.579	.398	2.16	.69	4.91	0.31
	Bag		13.03	5.05	.043	.242	.280	1.93	.34	6.05	0.71
18	Tins	Dry	12.01	5.05	.042	.228	.271	1.91	.47	6.02	0.73
		Normal	16.34	7.59	.042	.594	.247	2.11	.66	5.02	0.36
		Moist	18.14	7.12	.048	.567	.238	2.25	.75	5.01	0.31
	Bag		13.37	4.99	.041	.238	.279	1.84	.42	5.95	0.69
22	Tins	Dry	11.87	4.89	.037	.223	.234	2.19	.42	6.02	0.72
		Normal	16.23	9.10	.045	.855	.234	2.51	1.01	4.59	0.28
		Moist	18.22	5.90	.041	.373	.263	1.95	.45	5.10	0.38
	Bag		13.58	4.74	.040	.224	.267	2.14	.41	5.91	0.64

TABLE V—*Continued*

Period of storage			Moisture when sampled	Soluble extract	Soluble P_2O_5	Soluble nitrogen	Potassium sulphate soluble N	Maltose	Titratable acidity	pH	Buffer value
Weeks			%	%	%	%	%	%	%		
0			14.1	4.90	0.044	0.223	0.265	1.96	0.26	6.11	0.73
26	Tins	Dry	11.61	4.83	.041	.227	.230	1.43	.37	6.02	0.72
		Normal	16.02	8.27	.043	.790	.207	2.10	1.08	4.43	0.22
		Moist [1]	18.26	4.76	.041	.246	.232	1.95	.30	5.76	0.55
	Bag		12.80	4.70	.038	.221	.261	1.54	.34	5.93	0.67
30	Tins	Dry	10.53	—	—	—	—	—	—	6.05	0.81
		Normal	16.12	9.87	.054	.952	—	2.30	1.14	4.37	0.26
		Moist	18.33	—	—	—	—	—	—	6.48	1.03
	Bag		13.46	4.84	.048	.222	—	1.64	.48	5.89	0.79
34½	Tins	Dry	—	—	—	—	—	—	—	—	—
		Normal	16.78	7.38	.045	.563	.227	2.42	.61	4.99	0.40
		Moist	—	—	—	—	—	—	—	—	—
	Bag		13.21	4.80	.051	.226	.242	1.64	.47	5.95	0.73
39	Tins	Dry	11.45	—	—	—	—	—	—	—	—
		Normal	16.30	—	—	—	—	—	—	5.73	0.72
		Moist [2]	18.86	—	—	—	—	—	—	—	—
	Bag		12.84	—	—	—	—	—	—	6.04	0.82
49½	Tins	Dry [3]	12.00	4.88	.048	.231	.264	1.67	.43	—	—
		Normal [4]	16.68	5.10	.054	.247	.252	2.02	.37	—	—
		Moist [5]	19.23	3.97	.039	.211	.231	1.82	.24	—	—
	Bag [3]		13.64	4.78	.043	.226	.261	1.56	.48	5.88	0.59
60	Bag only		13.40	4.75	.044	.221	.253	1.60	.50	6.00	0.81
66	Bag only		13.15	4.75	.041	.231	.230	1.55	.48	5.99	0.80
78	Tins—Normal only		16.73	4.50	.035	.169	—	2.04	.31	6.46	.92
	Bag		13.60	4.77	.046	.233	—	1.65	.54	6.87	.53

[1] Slight unpleasant smell. [2] Strong smell of moulds. [3] No marked smell. [4] Very slight unpleasant smell. [5] Very strong mouldy smell.

TABLE VI

EFFECTS OF STORAGE AT DIFFERENT MOISTURE CONTENTS ON CHEMICAL COMPOSITION OF FLOUR. MANITOBA LOW GRADE
(*Results Calculated to 16% Moisture Basis*)

Period of storage			Moisture when sampled	Soluble extract	Soluble P_2O_5	Soluble nitrogen	Potassium sulphate soluble N	Maltose	Titratable acidity	pH	Buffer value
Weeks			%	%	%	%	%	%	%		
0			14.3	6.35	0.173	0.275	0.413	2.05	0.37	6.41	0.36
6	Tins	Dry	11.86	6.59	.183	.297	.432	2.29	.39	6.36	0.31
		Normal	16.30	6.32	.165	.293	.419	1.91	.46	6.25	0.30
		Moist	18.37	6.26	.205	.268	.341	2.26	.63	5.50	0.27
	Bag		12.85	6.56	.192	.290	.424	2.03	.41	6.34	0.30
10	Tins	Dry	11.71	6.39	.179	.280	.423	2.19	.40	6.33	0.30
		Normal	16.49	6.32	.181	.281	.462	1.95	.46	6.13	0.33
		Moist	18.29	6.74	.196	.278	.362	2.36	.81	5.31	0.27
	Bag		12.56	6.47	.182	.307	.392	2.30	.39	6.34	0.31
14	Tins	Dry	11.67	6.20	.178	.282	.423	2.10	.55	6.33	0.30
		Normal	15.80	7.10	.194	.309	.359	2.20	1.00	5.26	0.26
		Moist	18.80	6.60	.198	.275	.368	2.28	.77	5.30	0.22
	Bag		12.97	5.43	.153	.235	.338	2.03	.58	6.30	0.31
18	Tins	Dry	11.82	6.33	.175	.275	.419	2.0	.60	6.32	0.35
		Normal	16.28	7.83	.150	.372	.289	2.3	2.20	4.61	0.15
		Moist [1]	19.39	5.36	.138	.238	.355	2.29	.54	5.99	0.36
	Bag		12.96	6.11	.138	.271	.425	2.03	.65	6.31	0.34
22	Tins	Dry	11.57	6.39	.174	.273	.437	1.80	.61	6.28	0.30
		Normal	16.62	7.72	.155	.346	.280	2.32	1.88	4.57	0.14
		Moist [2]	20.15	5.40	.095	.385	.339	2.00	.26	6.40	0.36
	Bag		13.00	6.14	.185	.273	.406	1.93	.71	6.25	0.26

TABLE VI—*Continued*

Period of storage			Moisture when sampled	Soluble extract	Soluble P_2O_5	Soluble nitrogen	Potassium sulphate soluble N	Maltose	Titratable acidity	pH	Buffer value
Weeks			%	%	%	%	%	%	%		
0			14.3	6.35	0.173	0.275	0.413	2.05	0.37	6.41	0.36
26	Tins	Dry	11.59	6.41	.169	.264	.399	1.80	.54	6.33	0.34
		Normal [2]	16.77	7.10	.152	.310	.280	2.82	1.61	4.62	0.16
		Moist [4]	19.32	4.96	.068	.357	.379	2.71	.22	6.41	0.37
	Bag		13.07	5.79	.141	.256	.387	1.45	.69	6.30	0.29
30	Tins	Dry	11.38	—	—	—	—	—	—	6.35	0.38
		Normal	16.43	6.80	.167	.295	—	2.42	1.23	4.94	0.27
		Moist [8]	—	—	—	—	—	—	—	7.45	0.11
	Bag		13.53	6.01	.164	.250	—	1.75	.77	6.27	0.30
34½	Tins	Dry	—	—	—	—	—	—	—	—	—
		Normal	17.59	5.32	.105	.251	.343	2.45	.61	5.94	0.41
		Moist [7]	—	—	—	—	—	—	—	—	—
	Bag		13.50	5.88	.138	.260	.403	1.75	.81	6.23	0.38
39	Tins	Dry	11.75	—	—	—	—	—	—	—	—
		Normal	15.54	—	—	—	—	—	—	6.44	0.49
		Moist [7]	—	—	—	—	—	—	—	—	—
	Bag		13.02	—	—	—	—	—	—	6.24	0.38
49½	Tins	Dry [5]	12.13	6.02	.170	.266	.402	1.67	.74	—	—
		Normal [2]	18.14	5.05	.088	.217	.346	2.16	.38	—	—
		Moist [5]	—	—	—	—	—	—	—	—	—
	Bag [5]		13.60	5.85	.139	.263	.395	1.75	.92	6.07	0.25
60	Bag only		13.28	6.06	.156	.236	.392	1.75	.86	6.17	0.33
66	Bag only		13.21	5.97	.150	.258	.360	1.65	.86	6.15	0.32
78	Tins—Normal only		17.55	4.63	.080	.222	—	2.02	.31	6.87	.53
	Bag		13.50	5.96	.162	.257	—	2.23	1.09	6.19	.36

[1] Smell of moulds first noted.
[2] Slight unpleasant smell.
[3] Strong smell of moulds.
[4] Very strong smell.
[5] No marked smell.
[6] Heavy growth of moulds.
[7] Decomposing.
[8] Sample quite decomposed.

TABLE VII

EFFECT OF STORAGE AT DIFFERENT MOISTURE CONTENTS ON AMINO-ACID CONTENT AND PROTEOCLASTIC ACTIVITY OF FLOURS. ENGLISH PATENT FLOUR

Period of storage—weeks	Tinned samples									Bagged sample		
	Dry			Normal			Moist					
	Initial amino-acid content	Amino-acid after 2 hrs. incubation	Increase = proteo-clastic activity	Initial amino-acid content	Amino-acid after 2 hrs. incubation	Increase = proteo-clastic activity	Initial amino-acid content	Amino-acid after 2 hrs. incubation	Increase = proteo-clastic activity	Initial amino-acid content	Amino-acid after 2 hrs. incubation	Increase = proteo-clastic activity
	%	%	%	%	%	%	%	%	%	%	%	%
2	0.094	0.130	0.036	0.091	0.129	0.038	0.092	0.129	0.037	0.099	0.142	0.043
6	0.092	0.131	0.039	0.090	0.126	0.036	0.096	0.134	0.038	0.098	0.134	0.036
10	0.096	0.135	0.039	0.084	0.139	0.055	0.134	0.221	0.087	0.078	0.140	0.062
14	0.099	0.124	0.025	0.097	0.127	0.030	0.147	0.220	0.073	0.090	0.116	0.026
18	0.092	0.118	0.026	0.091	0.114	0.023	0.088	0.129	0.041	0.086	0.110	0.024
22	0.120	0.140	0.020	0.118	0.148	0.030	0.124	0.130	0.006	0.092	0.132	0.040
26				—	—	—				—	—	—
30				—	—	—				—	—	—
34½				0.171	0.239	0.068				0.090	0.140	0.050
39				0.235	0.294	0.059				0.100	0.146	0.046
49½										0.096	0.134	0.038
60										0.107	0.140	0.037
66										0.098	0.140	0.042
78										0.113	0.159	0.046

TABLE VIII

EFFECT OF STORAGE AT DIFFERENT MOISTURE CONTENTS ON THE DRIED GLUTEN CONTENT OF FLOURS

Period of storage—Weeks	English Patent				English Low Grade				Manitoba Patent				Manitoba Low Grade			
	Tinned samples			Bagged sample	Tinned samples			Bagged sample	Tinned samples			Bagged sample	Tinned samples			Bagged sample
	Dry	Normal	Moist		Dry	Normal	Moist		Dry	Normal	Moist		Dry	Normal	Moist	
	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%
6	8.7	8.8	8.3	8.7	8.5	8.5	7.3	8.5	12.6	12.5	11.7	12.0	13.9	14.3	12.4	13.8
10	8.5	8.6	6.7	8.8	8.4	8.5	7.5	8.4	11.9	11.9	10.3	12.1	14.0	13.3	12.9	14.2
14	8.4	8.8	5.9	8.8	8.4	8.4	7.3	8.4	12.0	12.1	10.2	12.1	13.5	12.4	12.1	13.8
18	8.5	8.7	7.9	8.5	8.4	8.3	7.5	8.4	12.2	10.5	10.2	12.2	14.0	12.1	12.5	13.6
22	8.7	8.7	8.5	9.0	8.6	8.1	8.0	8.6	12.2	9.3	11.4	12.4	14.2	12.4	12.5	13.7
26	8.7	8.6	8.5	9.0	8.7	7.4	7.7	8.4	11.9	8.8	11.8	12.1	14.1	12.2	nil	13.4
30	8.8	7.1	8.5	8.4	8.5	6.6	nil	8.5	11.6	8.8	10.7	11.9	13.4	12.1	—	13.1
34½	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
39	8.4	nil	8.1	8.5	8.5	5.6	—	8.2	11.8	11.2	10.3	11.7	13.2	12.6	—	12.7
49½	8.8	nil	7.7	8.7	8.4	6.3	nil	8.2	11.7	11.6	nil	11.6	13.5	13.2	—	12.9
60	8.7	—	—	8.7	—	—	—	8.4	—	—	—	12.0	—	—	—	13.6
66	—	—	—	8.5	—	—	—	8.4	—	—	—	11.8	—	—	—	14.2
78	—	nil	—	8.6	—	7.0	—	8.4	—	11.1	—	11.8	—	12.7	—	13.5

with all four flours; the maxima were of much smaller magnitude than those found with the normal samples but occurred earlier, *e.g.*, after 1 to 4 months.

Precisely similar types of change occurred in pH and buffer values.

Nitrogen Changes During Storage

The changes in soluble extract and in soluble nitrogen showed some irregularities and differences. In amounts of total soluble matter and of soluble nitrogen, the Manitoba changed less than the English and the low-grades less than the patents, the reverse of what took place with the acidities. The dry samples—both bagged and tinned—showed no appreciable alterations in soluble extract and soluble nitrogen throughout the whole period of storage. The normal and moist samples showed the same type of periodic variation as was shown by the acidities and pH's. That is, the normal samples showed slow increases for some months followed by rapid increases to maximum values, which with the Manitoba samples were followed by rapid decreases. The moist samples showed rapid increases in the earlier months of storage followed by equally rapid decreases, after which the values remained stationary or decreased very slowly, except with the English low-grade in which both soluble extract and soluble nitrogen increased again. In both soluble extract and soluble nitrogen the two low-grades showed markedly less change than the patent; the Manitoba low-grade (all four samples) showed comparatively little change in soluble nitrogen.

Reference to the tables will indicate that although there are some irregularities in the percentages of soluble phosphorus, nitrogen soluble in potassium sulphate solution, and in maltose figures, no pronounced progressive changes took place in these. The constancy of the potassium sulphate soluble nitrogen is interesting in view of the large variations in the water soluble nitrogen, as is also the constancy of the soluble phosphorus in view of the great changes in acidity.

Proteolytic Activity

The changes in amino-acid content and in proteoclastic activity were similar to those observed in acidity. The bagged and dry samples changed very little although showing some irregularities, while the normal and moist samples showed the same striking periodicities. To save space, the figures for one flour only are given. These determinations were carried out by the method of Denham and Scott Blair (1927).

Discussion

There can be little doubt that many of these surprising changes in chemical properties of flour during storage must be due to bacterial or

fungal action, or to both. Biological activities are more marked and more rapid the higher the moisture content of the material, and the rhythmic character of many of the characteristic changes during storage, which does not appear to have been noticed by previous workers, is certainly suggestive of biological action. Bacterial activities are very sensitive to changes of acidity of the medium. Bacteria do not flourish in markedly acid surroundings, a pH of about 4 being quite inimical to most bacteria; moulds, on the other hand, are more resistant. The results suggest that the earlier changes may be, in part at least, due to bacteria, some mould action and enzymic changes doubtless proceeding at the same time. As the acidity approaches a pH of 4, mould action with consequent production of ammonia and reduction of acidity may become predominant. Certainly, the smells that developed in the moist samples after 5 to 7 months storage were all characteristically "mouldy," and it is significant that in no case did a smell develop until the acidity had passed the maximum and had returned to almost its original value or below.

The immediate practical question that arises, from a consideration of the results already discussed, is whether these rhythmical changes in chemical properties can be correlated with any similarly rhythmical changes in baking quality of the flours. This question can perhaps be approached best by considering the characters of the periodically washed-out glutens, the percentages of which are given in Table VIII.

The glutens from the bagged and tinned dry samples of English patent and low-grade flours showed very little change either in quality or quantity, except the tinned dry low-grade which showed a slight gradual improvement up to the 10th month followed by a bad falling-off in quality, but not in quantity, at the 12th month. The tinned normal samples showed little change, either in quantity or quality, for 7 months (patent) and 5 months (low-grade) respectively. The patent then showed a sudden breakdown in quality and reduction in quantity (from 8.6% to 7.1%) at the 8th month, and no gluten at all was obtainable at the 10th and 12th months. The low-grade showed a bad and progressive falling-off in quality and in quantity (from 8.3% to 5.6%) between the 5th and the 10th month, followed by a distinct improvement at the 12th month. The tinned moist English samples were of great interest. With the patent a sudden marked and progressive break-down occurred at the 3rd and 4th months, followed by a marked and progressive improvement at the 5th and 6th months, which was then followed by a very slow falling-off in quality and quantity from the 7th to the 12th month. At the same time the gluten at the 6th month was inferior in quality to, and of rather different character from, that obtained at the 1st and 2nd months; that is, the second improvement did not bring the

gluten back to its original quality. The tinned moist low-grade showed a steady improvement up to the 3rd month, then a steady deterioration to the 6th month; the 7th month was of very poor quality and no gluten was obtained at the 8th, 10th, and 12th months.

The Manitoba patent, tinned dry sample, showed a gradual slight improvement to the 4th, 5th, and 6th months, which were similar, and a gradual falling-off in quality from the 6th to the 12th month. The bagged patent sample showed little change in quality for 13 months, the sample at the 14th month being distinctly poorer. The tinned normal patent sample showed gradual improvement in quality to the 3rd and 4th months, then a progressive falling-off in quality and quantity (from 12.1% to 8.8%) to the 7th month, which was very poor, and finally a marked and progressive improvement in quality and quantity (from 8.8% to 11.6%) to the 12th month. The tinned moist sample showed gradual deterioration after 2 months; this deterioration increased rapidly, the gluten quality being very poor at the 8th and 10th months; no gluten at all was obtained at the 12th month.

The tinned dry and bagged samples of low-grade Manitoba behaved alike. Both showed gradual improvement to the 3rd month; there was little further change to the 7th month, and a sudden and pronounced fall in quality at the 8th month. The glutens of the 8th and subsequent months were all similar and very poor. The tinned normal sample showed no change for the first 2 months; a sudden drop in quality occurred at 3 months and was progressive to the 6th month. The 6th, 7th, and 8th months' glutens were similar in quality while those at the 10th and 12th months were slightly better. The tinned moist sample showed a steady deterioration in quality, although not in quantity, up to the 6th month, after which no gluten was obtainable.

It is evident that the periodic changes in many chemical properties of flours during storage are accompanied by somewhat similar changes in gluten characters and similar periodic changes might be expected in baking characters. It has not been easy to investigate this point. The changes were most pronounced in the tinned samples with the higher moisture contents, but the samples were too small to permit baking tests being carried out. Baking tests were carried out only on the large bagged samples, and in these (on account of their relatively low moisture contents) the changes during storage were relatively slow, with, in 18 months, little indication of any periodicity.

The baking tests were carried out on the multiple differential system described elsewhere (Fisher and Halton, 1936). Two per cent yeast, 1¼% salt, the necessary water, and a dough temperature of 80° F. were employed, and all doughs of any one series went to the oven together, representing 1¾, 2½, 3¼, 4, and 4¾ hours fermentation (including

final proof of ¾ hour). No very marked changes in dough or loaf characters were observed during 18 months. The Manitoba samples improved somewhat in dough "body," and the English, after some months, showed a somewhat increased dough tenderness and "shortness," but fermentation time and tolerance and general loaf characters showed no marked changes in any of the flours.

Indirect evidence, however, has been obtained which suggests very strongly that the rhythmical changes noticed in chemical characters during storage would be reflected in similar changes in baking characters. It has already been mentioned that, of two samples of No. 1 Manitoba and ordinary English flours stored for 2 years, the Manitoba flour had improved in baking quality by the end of that time, whereas the English flour had become thoroughly broken down, the resulting loaf being small and most unappetising. Photographs of the cut loaves are given in Figure 1. At the end of 4 years' storage the same sample of Manitoba flour showed comparatively little further change in baking quality, whereas the same sample of English flour had very materially improved. The English loaf was still small and rather unappetising, but all dough and loaf characters showed very marked improvement over the 2 years' old sample. Photographs of the two cut English loaves are shown in Figures 1 and 2.

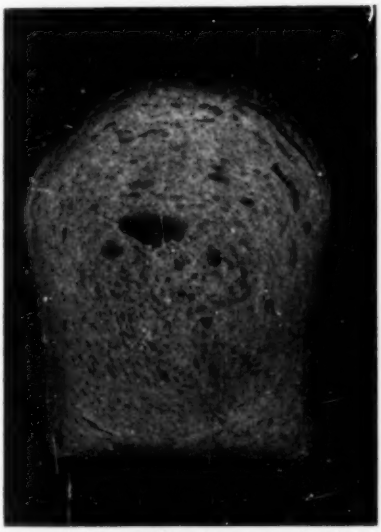

Fig. 2. Cross-section of loaf made from portion of same sample of English flour as in Figure 1, but after 4 years' storage.

Improving Effect of Aged Flours

Another peculiarity of very old flour was discovered during the course of this investigation. It has already been stated that the use

of chemical improvers arose from the desire of millers to expedite or to imitate the improvement brought about slowly during storage. Of recent years attempts have been made to effect these necessary improvements by physical rather than by chemical means. In this country two groups of physical processes have been developed commercially, *viz.*, those patented by D. W. Kent-Jones and Woodlands, Ltd. (1925, 1925a, 1929), and by E. A. Fisher, C. R. Jones, and the Research Association of British Flour-Millers (1928). It was first shown by Robert Hutchinson (1924) that the marked improvement brought about by limited heat-treatment under certain conditions is followed by marked and progressive deterioration when the heat treatment is prolonged, until a point is reached when no gluten can be washed from the flour, and the flour itself is no longer fit for bread-making. Such over-treated flour, however, when added to untreated flour to the extent of 15% (= 42 lb. per sack) brought about a marked improvement in baking quality. This idea was developed further by D. W. Kent-Jones and Woodlands Ltd. (1925), who showed that, by over-treating to a still greater extent, the amount of such over-treated flour required to effect maximum improvement in untreated flour could be reduced to about 0.7% (= 2 lb. per sack).

It occurred to the writers that flour, which had become seriously damaged and useless for bread-making by prolonged storage, or by less prolonged storage under unsatisfactory conditions, might have improving action when added to untreated flour. This was found to be so. The English flour referred to above, which, after 4 years' storage, was quite unfit for bread-making, effected a very striking improvement in a straight run untreated London commercial flour when added to the latter to the extent of 2%. The improvements resulting were improved dough body and stability, improved oven spring, and improved crumb colour, softness and spring, and were quite comparable with those brought about in the same flour by subjecting the whole flour to either of the patented heat-treatment processes referred to above. Photographs of the cut loaves are given in Figures 3 and 4. A number of other very old flours have been examined with similar results; all effected improvements to varying degrees when added to the extent of 2% to ordinary commercial untreated flours.

It has been reported by millers that this phenomenon is known to the industry, although it was unknown to the writers when these experiments were conducted. It is obvious that if systematic use could be made of this knowledge every miller could manufacture his own improver and so be independent of the manufacturers of chemical and physical improvers and improvement processes, and at merely nominal cost to himself. At present, however, we have no knowledge concern-

ing the precise nature of the changes concerned; we do not know the precise point of development of the old flour at which this improving action is at a maximum; and we have no means of determining this point except by periodical baking tests of every sample stored. Moreover, it would probably be impracticable—if only on account of lack of warehouse accommodation—for a miller to store 2% of his output of flour for 2 or more years in order to make his own improver on the

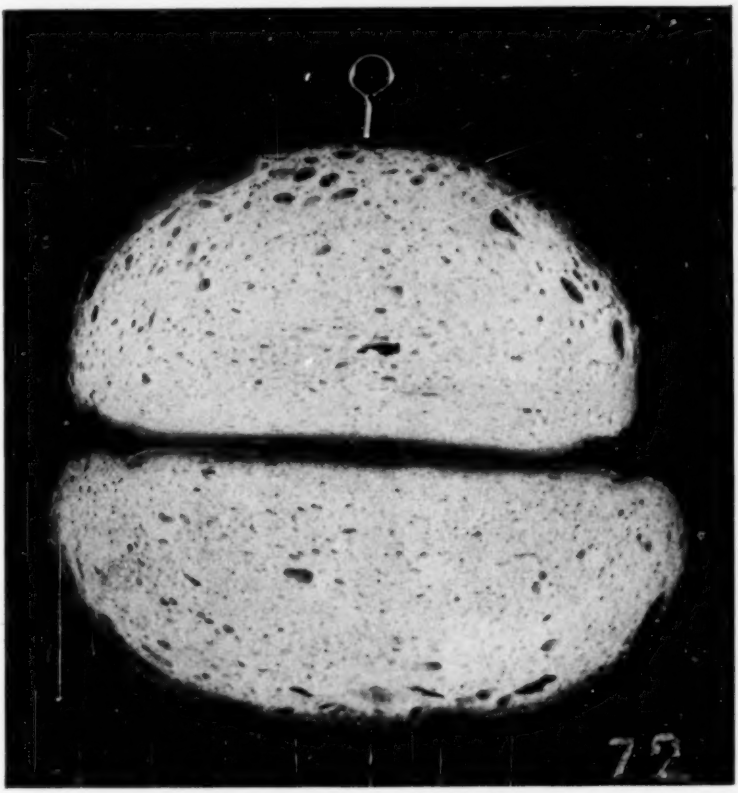

Fig. 3. Cross-sections of loaves made from commercial straight run flour. Bottom loaf from untreated flour, top loaf from same flour with the addition of 2% of 4-year old flour.

premises. It seems necessary to reduce the time required for the change, or, alternatively to effect the changes in a shorter time by some other means. Again, different flours respond differently, particularly as regards rate of change, to storage, and it is probable that different samples of nominally the same flour, *e.g.*, English, would show marked differences in this respect. English flours from different parts of the country, or milled in different mills, or from different crops would almost certainly show a wide range in bacterial and fungal content, and

would be expected to show marked differences in their response to storage. One of the biggest practical difficulties would be to turn out a really standardised product. This variability in improving action is shared alike by over-stored and by over-heat-treated flours; in neither case has it proved possible so far to manufacture such an improver of really regular character. Many samples of over-stored flour and of over-

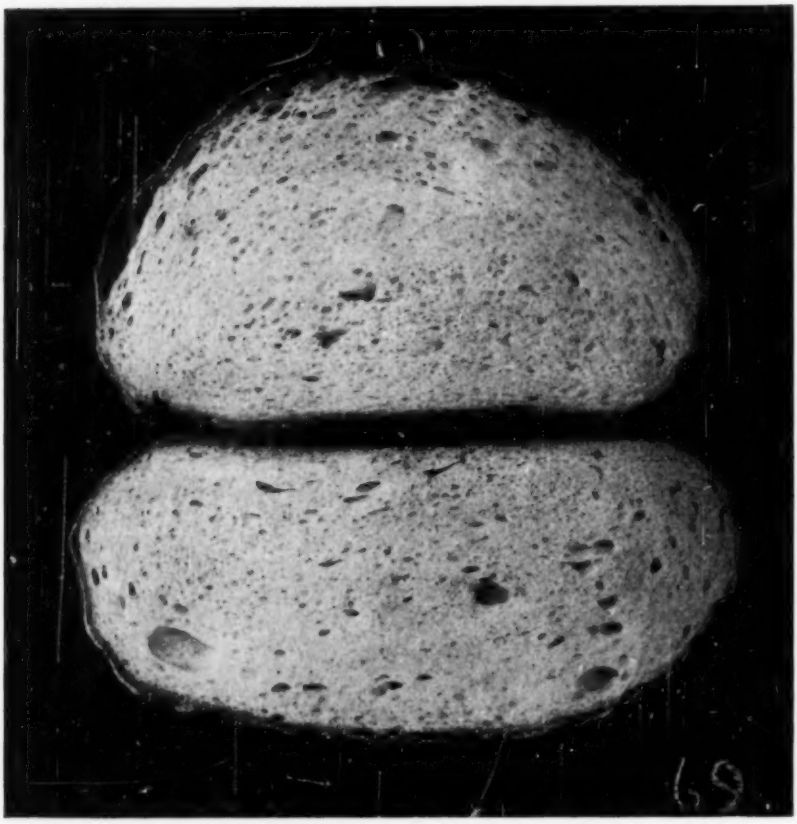

Fig. 4. Cross-sections of loaves made from same flour as in Figure 3. Bottom loaf from untreated flour, top loaf from heat-treated flour.

heat-treated flour have been examined in the writers' laboratories, and wide differences in improving action have been observed in both types; some samples have given eminently satisfactory results, others of nominally the same type have been of little value. It seems unlikely that these difficulties will be overcome until exact knowledge has been acquired of the nature of the changes concerned.

Summary

An extensive investigation is described of the changes taking place in flour during 18 months' storage. Four flours were studied—an ordinary English (non-Yeoman) patent (60%) and low-grade (40%), and a No. 3 N. Manitoba patent and low-grade. Samples of each flour at three different moisture contents—approximately 12%, 16%, and 18% respectively—were stored in closed tins, the remainder of each sample being stored in its original bag as received. In addition to baking tests on the bag samples the following analytical determinations were carried out on the sixteen flour samples, at first at monthly and later at bi-monthly intervals: moisture, soluble extract, soluble phosphorus (P_2O_5), soluble nitrogen, nitrogen soluble in 5% potassium sulphate solution, maltose figure, dried gluten (by washing out), total or titratable acidity, hydrogen ion concentration, buffer value, amino-acid content, and proteoclastic activity.

The detailed results are given in Tables III to VIII.

It is well known that all flours improve in baking quality during storage up to a point, beyond which deterioration sets in. This deterioration, it has been stated, continues until the flour is entirely unfit for bread-making (see Figure 1). Moreover, the initial improvement may continue to increase for several years in the case of high grade Manitobas while with soft flours like English a few months may be the limit of safe storage. The results described indicate that the story is much more complicated than this. After the first deterioration has proceeded for some time a second improvement sets in (see Figure 2), which in turn may be followed by a second deterioration. A similar periodicity occurs in changes observable in many of the chemical properties, among which may be mentioned hydrogen ion concentration, buffer value, total acidity, soluble nitrogen and, above all, in amount and quality of the washed out gluten. These changes are much more marked and take place more rapidly the higher the moisture contents of the flours, and also show very marked differences according to the nature and grade of the flour. It is suspected that biological activities, due to moulds and bacteria, play important parts in these changes.

Flour which has become useless for bread-making by prolonged storage, or by less prolonged storage under unsatisfactory conditions, has a marked improving action when added at the rate of 2% to otherwise untreated flour. The improvements resulting are improved dough body and stability, improved oven spring, and improved colour, softness and spring of crumb, and are quite comparable with those brought about by heat treatment processes, or by the use of over-heat-treated flours (see Figures 3 and 4). The precise point of development of the old

flour at which this improving action is at a maximum, the length of storage required for this point to be reached, how that time can be reduced to periods short enough for practical advantage to be taken of these changes, and above all the precise nature of the changes are important problems which must be solved before systematic use can be made of the information so far available.

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DIASTATIC ACTIVITY IN CONNECTION WITH FLOUR MILL CONTROL

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A great deal of work has been done in connection with the diastatic activity of wheat flour. The comparatively recent development of correcting the diastatic activity at the mill by the proper addition of small amounts of malted wheat flour, commands our attention. It is our purpose to bring out some facts of interest in this connection. In order to do this, it was found necessary to repeat the work of a number of previous investigators. The objective of our study was to develop some knowledge which might be of practical value in the diastatic adjustment of flour by the addition of malted wheat flour as is now commonly practiced.

Previous Investigations

Diastatic adjustment.—Dombach (1912) patented a process for improving flour quality by the addition of germinated wheat to the wheat mixture. Sherwood and Bailey (1926) conducted extensive experiments with germinated wheat and demonstrated the increase of diastatic activity of the resultant flour milled from mixtures of this wheat with ungerminated wheat.

Variety and climate, rainfall, etc.—Mangels (1926) working with three varieties of wheat grown at different stations showed the three to have inherently different diastatic properties and that the pre-harvest rainfall and temperature exerted a marked influence. In another paper

(Mangels, 1926a), using commercial starch as a substrate, he states, "The variation in diastatic activity of flour appears to be due in large part to the susceptibility of the starch granule to diastase rather than the concentration of diastase present." Mangels also demonstrated that soil fertility had a bearing on it. Bracken and Bailey (1928) found that no change in diastatic activity occurred as a result of leaving the wheat uncut in the field after once ripe. Hermano and Rask (1926) worked on the susceptibility of different wheat starches and indicated that individual variations might be traced to geographical origin and climatic variations of the wheat. Swanson (1935) found diastatic activity varies in different classes of wheat; those grown under very dry conditions were lower than those grown under more moist conditions.

Granulation, degree of fineness, and starch susceptibility.—Alsberg and Griffing (1925) and Alsberg (1927) found that overgrinding of flour resulted in increased diastatic activity. Alsberg (1927) concluded that the fermentation rate of a flour must depend upon at least three factors: the diastase content, the content of uninjured granules, and the susceptibility to diastase attack of the uninjured granules. Pascoe, Gortner, and Sherwood (1930) state, "The degree of granulation of flours is an important factor in the consideration of their saccharogenic properties. . . ." Karacsonyi and Bailey's (1930) work led to the conclusion that "the saccharogenic activity of an aqueous flour suspension is substantially increased by overgrinding the flour . . . , and seems to indicate that if the saccharogenic activity is comparatively low, the increase is more significant than if the value is higher." Hermano and Rask, in their aforementioned work on the susceptibility of different wheat starches, indicated a variation due to the condition of the starch. Markley and Bailey (1934) found that the particle size was not a major factor in determining the diastatic activity of flour. Their work showed that grinding in an atmosphere of 50% humidity gave higher average diastatic activity than grinding in an atmosphere of 75% humidity.

Effect of flour grade.—Kalning and Schleimer (1913) found the Lintner value to increase as the grade of flour decreased and that diastatic activity paralleled the fat content. These authors considered the Lintner value as a chemical criterion of the proportion of germ in the flour. Martin (1920) found the flour from the interior of the wheat berry to be the lowest in diastatic activity. The flour, exterior to the interior flour, was the next lowest in diastatic activity. The flour in the region near the cortex had the next highest diastatic activity, while the flour which had the highest diastatic activity of all came from the region closest to the cortex. Bailey (1925) states that the activity of the disintegrated germ and aleurone should be greater than in the pulverized endosperm. Pascoe, Gortner, and Sherwood (1930), in a test of mill

streams, found, in general, that "the high values for the saccharogenic activity are found in the lower grades of flour," and that this high activity of the low grade, duster flour, sizings, first and second tailings, fourth and fifth breaks, may be attributed to relatively high germ content.

Experimental milling.—Pascoe, Gortner, and Sherwood (1930), comparing experimentally and commercially milled flours from the same wheat, found the commercially milled flours in all cases to have a higher saccharogenic activity than the experimentally milled flours, and they state that "it is apparent that flours milled with the laboratory mill do not provide reliable information regarding the saccharogenic properties of commercially milled flours from the same wheat," and they believe "the difference in size of particles remain as the probable principal casual factor." Markley and Bailey (1934) found the diastatic activity of experimentally milled flour to be correlated with the diastatic activity of flour milled in a commercial mill from aliquots of the same wheat. In all cases, the experimentally milled flours were lower. Swanson (1935) also found the experimentally milled flours to be lower.

Plan of Experiments

The purpose of this work was to learn something about the diastatic reaction of various flours to treatment with diastatic malted wheat flour. The experiment was divided into five parts:

(*A*) Analysis and treatment of a number of flours from widely different localities and ground from different varieties of wheat.

(*B*) Analysis and treatment of the flour streams from an 1800-barrel commercial mill, grinding Kansas hard winter wheat of 12.75% protein content.

(*C*) Analysis and treatment to bring out from a practical mill operative's standpoint, the effect of differences in particle size or granulation.

(*D*) Investigation of the practicability of using the experimental mill in estimating the proper malt treatment to give flour made from the same wheat on a commercial mill.

(*E*) Method for comparing the strength of different malt flours and the amount to use to obtain any desired maltose content.

In all cases of malt treatment, ¼ of 1% of a standard pure malted wheat flour which had been thoroughly blended was used. This malt flour was kept, throughout the experiment, in an air-tight container under refrigeration.

Methods of Analyses Used

The method of Blish and Sandstedt (1933) was employed throughout this work to determine diastatic activity. Collaborative work by

C. F. Davis (private communication) and others indicates that the Blish method gives results slightly higher in the range between 150 mg. and 200 mg. maltose, and low above the range of 250 mg. maltose, as compared with the average of all methods studied. However, it is believed that the Blish-Sandstedt method is accurate enough for all practical purposes, and has the advantage that a great number of tests may be run at one time and results duplicated very accurately. The Rumsey maltose blank was determined by the simplified method of Blish and Sandstedt, using 0.4% sulphuric acid previously cooled in ice-water. All maltose values are the average of replicates which were not accepted unless they agreed within 3 mg. maltose. All values reported throughout the work are on the basis of 15% moisture and are the average of duplicates or more.

The work of Jørgensen (1931), Larmour, Geddes, and Whiteside (1933), and Davis and Worley (1934) all show a high correlation between diastatic activity and gassing power. The latter work shows that for the practical purposes of mill control, we are justified in concluding that the dough gas production will vary approximately as the maltose values as determined by the method of Blish and Sandstedt (1933). Blish, Sandstedt, and Astleford (1932) conclude that the terms "diastatic activity" and "gassing power" are not strictly synonymous. Lack of parallelism between these two properties is due to variations among flours with respect to original sucrose content.

Blending of the malt flour and the sample of flour was accomplished by weighing out 100 g. of the flour sample, and introducing it along with the malt flour into a small mechanically driven blender. This blender revolved at a speed of 7 r.p.m. for 5 minutes and had a series of three baffles in each opposite side, so that the sample was divided into four parts at each one-half revolution, and thrown across diagonally to the opposite end. An eccentric jars the mixer vigorously at each one-half revolution, and mixing is very thorough and complete.

The ether extract results were secured on a 16-hour extraction in duplicate on 2 g. of flour, 15% moisture basis, without previous drying. It was found impossible to obtain checks on replicates if the sample was first dried. Greville (1923), Kent-Jones (1924), and Herd (1927) pointed out that preliminary drying gives much lower results.

Experimental

(*A*) Analysis and Treatment of Different Types of Flours

Unbleached patent, clear, and low grade flours were secured from mills in a number of different localities grinding different varieties of wheats grown under varying conditions of soil and climate. Results obtained are shown in Table I.

TABLE I

DIASTATIC ACTIVITY AND RESPONSE TO MALT TREATMENT OF DIFFERENT TYPES OF FLOURS[1]

Mill No.	Class of wheat	Grade of flour	Ash	Protein (N×5.7)	Maltose blank	Maltose	Maltose (+¼% malt flour)	Maltose increase
			%	%	mg.	mg.[2]	mg.[2]	mg.
1	Western Turkey (1934)	90% Patent	0.437	12.03	28	186	267	81
	Utah-Idaho Turkey (1935)	90% Patent	.421	11.23	21	231	329	87
	Utah Turkey (1935)	10% Clear	.632	12.53	26	190	288	98
2	Texas hard winter	Patent	.401	12.39	27	206	292	86
		Clear	.628	14.67	27	183	258	75
		Low grade	.935	17.80	30	222	301	79
3	Canadian spring	Patent	.363	12.14	20	273	384	111
		Clear	.695	14.92	26	230	319	89
		Straight grade	.432	12.41	24	259	371	112
4	Indiana soft red winter	Patent	.336	7.48	21	99	152	53
		Clear	.384	9.06	19	109	165	56
		Low grade	.651	10.35	28	134	201	67
5	Pacific Coast soft white	Patent	.344	7.22	28	195	253	57
		Clear	.443	7.66	28	191	240	48
		Low grade	.617	8.53	27	210	269	59
6	North Dakota spring	Patent	.426	12.49	21	324	398	74
		Clear	.738	14.57	22	331	405	74
		Low grade	1.333	16.14	31	435	486	51
		97% Extraction	.465	12.62	22	326	399	73
7	Kansas hard winter	Patent	.392	11.06	20	185	266	81
		Clear	.536	12.47	21	179	256	76
		Low grade	.721	12.59	24	192	268	76
8	Kansas hard winter	Patent	.384	11.12	20	198	280	82
		Clear	.556	12.81	23	188	271	83
		Low grade	.770	13.18	21	216	299	83

[1] All results are in duplicate and on a 15% moisture basis.
[2] Milligrams of maltose from 10 g. of flour after 1 hour's diastasis at 30° C.

Maltose blank values do not appear to be significant, although there appears to be a tendency for the low grade flours to run slightly higher than the patent or clear flours.

Differences in initial diastatic activity due to class of wheat or geographical location are outstanding (see Table I). The highest results are from mill No. 6 grinding North Dakota spring wheat and mill No. 3 grinding Canadian spring wheat. Mills Nos. 2, 7, and 8, grinding hard winter wheat, are the next highest excepting mill No. 5, grinding Pacific Coast soft white wheat. Mill No. 4, grinding Indiana soft winter wheat, is much the lowest. Mill No. 5 shows results surprisingly high for a soft type of wheat.

The maltose increase from the malt flour treatment is not consistent either for class of wheat or grade of flour. Mill No. 3, grinding Canadian spring wheat with a high initial diastatic activity, showed a large response on the patent and the straight grade but the clear was considerably lower. The lowest response was exhibited by mill No. 4, grinding Indiana soft red winter wheat, and mill No. 5, grinding Pacific Coast soft white wheat. Examination showed the flour from mills Nos. 4 and 5 to be far the softest and finest of any in the series, and suggested that their starch would be more susceptible to conversion by the malt treatment. It is interesting to note that mills Nos. 2, 7, and 8, all grinding hard winter wheat, showed practically the same relative differences in initial diastatic activity between their three grades. In all three cases, the clear was the lowest, with patent next, and the low grade the highest. Their response to malt treatment ran fairly uniform among the three, being less than the spring wheat from Canada but greater than the North Dakota spring wheat and considerably greater than the soft red winter or the Pacific Coast white wheats. It is quite probable that the flour stocks in these mills are classified quite similarly between the patent, clear, and low grade.

The two 90% patents listed at the head of the column for mill No. 1 were made on the same mill but show a big difference in initial maltose content and a slight difference in response to malt treatment. On examination, the sample from the 1934 crop proved to be much more granular than that from the 1935 crop. The 10% clear flour made in mill No. 1 was considerably higher both in initial diastatic activity and response to treatment. This particular sample appeared to be very finely ground and made up of extremely soft stocks.

Examination of these results by reference to Table I shows the inherent differences in diastatic activity due to difference in class of wheat and the location in which they were raised. It would also indicate the difference in the initial diastatic activity of the flours, and the response of the flours to treatment might be due to the way the stocks are classified in the different mills, and the fineness of grinding.

(*B*) Analysis and Treatment of the Flour Streams from an 1800-Barrel Commercial Mill

The results secured in these tests are shown in Table II. The size silks through which this flour was bolted is shown at the bottom of the table. It should be mentioned, however, that grade of silk does not necessarily indicate the fineness of the flour bolted through it, since the size of the load on each particular section is a big factor in the freedom with which the stock bolts. It was found desirable to plot graphs in an attempt to determine the correlation between the different properties shown.

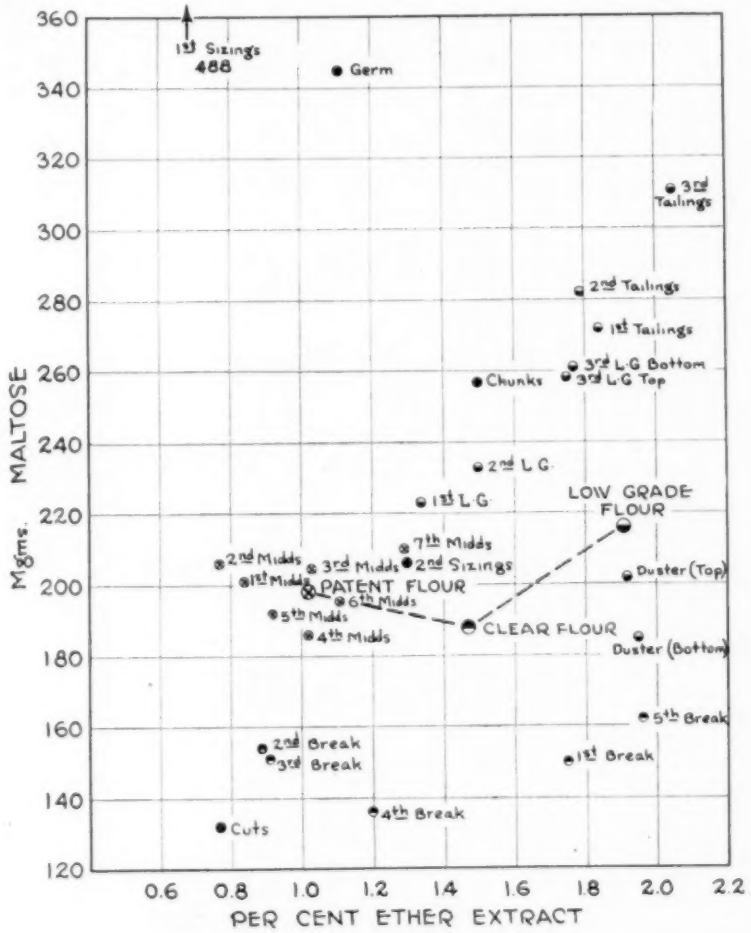

Figure 1. Correlation between maltose and ether extract.

In Figure 1 the maltose is plotted against the ether extract. Reference to the graph will show that the middlings fall within a rather

TABLE II

DIASTATIC ACTIVITY OF FLOUR MILL STREAMS FROM AN 1800-BARREL MILL
(Kansas Hard Winter Wheat—12.75% Protein) [1]

Flour stream	Ash	Protein (N×5.7)	Ether extract	Maltose blank	Maltose	Maltose (+¼% malt flour)	Maltose increase	Remarks
	%	%	%	mg.	mg.[2]	mg.[2]	mg.	
1st break	0.557	11.48	1.75	20	148	208	60	
2nd break	.544	11.13	0.89	20	155	220	65	
3rd break	.433	11.94	0.91	18	148	218	70	Break flour becomes finer and softer
4th break	.525	14.45	1.20	18	136	213	77	
5th break	.657	15.38	1.96	21	163	245	82	
Chunks	.470	10.34	1.49	22	257	347	90	Much germ through this
1st sizings	.411	9.91	0.69	22	488	608	120	Most of germ through this
2nd sizings	.463	10.96	1.30	21	206	277	71	
1st middlings	.362	10.52	0.84	20	201	272	71	
2nd middlings	.348	10.72	0.77	20	206	284	78	
3rd middlings	.408	11.33	1.03	22	205	281	76	
4th middlings	.393	11.44	1.02	22	186	269	83	
5th middlings	.391	11.29	0.92	22	192	273	81	
6th middlings	.391	11.32	1.11	22	196	280	84	
7th middlings	.400	11.03	1.29	22	211	296	85	
Cuts	.440	12.35	0.77	18	132	192	60	Middlings dustings from first three breaks—very sharp and hard
Germ	.460	9.99	1.11	22	345	433	88	Pure germ separated here

TABLE II—*Continued*

Flour stream	Ash	Protein (N×5.7)	Ether extract	Maltose blank	Maltose	Maltose (+¼% malt flour)	Maltose increase	Remarks
	%	%	%	mg.	mg.[2]	mg.[2]	mg.	
1st tailings	.697	11.20	1.84	26	272	346	74	
2nd tailings	.700	11.33	1.79	26	282	366	84	
3rd tailings	1.044	11.62	2.05	32	311	390	79	
1st low grade	.513	11.60	1.34	22	223	306	83	
2nd low grade	.603	12.77	1.50	23	233	314	81	
3rd low grade top	.810	12.51	1.75	27	259	350	91	
3rd low grade bottom	.914	13.29	1.77	32	261	350	89	
Duster top	.835	13.43	1.92	24	202	267	65	
Duster bottom	.839	13.52	1.85	25	196	260	64	
Patent [3]	.384	11.12	1.02	20	198	280	82	
Clear [4]	.556	12.81	1.46	23	188	271	83	
Low grade [5]	.770	13.18	1.91	25	216	299	83	
Bran	6.44	15.60 [6]	3.26	91	253			
Shorts	3.79	17.12 [6]	4.32	95	332			
Pure germ [7]	4.66	28.28 [6]	9.63	156	436			

[1] Mill clothed mostly with 11-XXX, 12-XXX silks, some 10-XXX, some 14-XXX, and a few 15-XXX.
[2] Milligrams of maltose from 10 g. of flour after 1 hour's diastasis at 30° C.
[3] Patent flour contained 1st to 7th middlings flour, 1st and 2d sizings, 2d and 3d breaks, and cuts.
[4] Clear flour contained 1st break, 4th break, 5th break, chunks, and 1st low grade.
[5] Low grade flour contained bran duster (top and bottom), 2d low grade, 3d low grade, and 3d tailings.
[6] Protein on bran, shorts, and germ is reported as N×6.25
[7] Pure germ was reground before ether extract, maltose blank, and maltose were determined.

close range, the breaks fall within a lower range, and the correlation between the low grade and tailings stock is quite good. The three flours, whose maltose value is much higher than their ash would indicate, are the first sizings, the germ flour, and the chunks, the first two being especially outside the range. The chunk stock is a very coarse middlings with considerable bran attached and some germ, and might

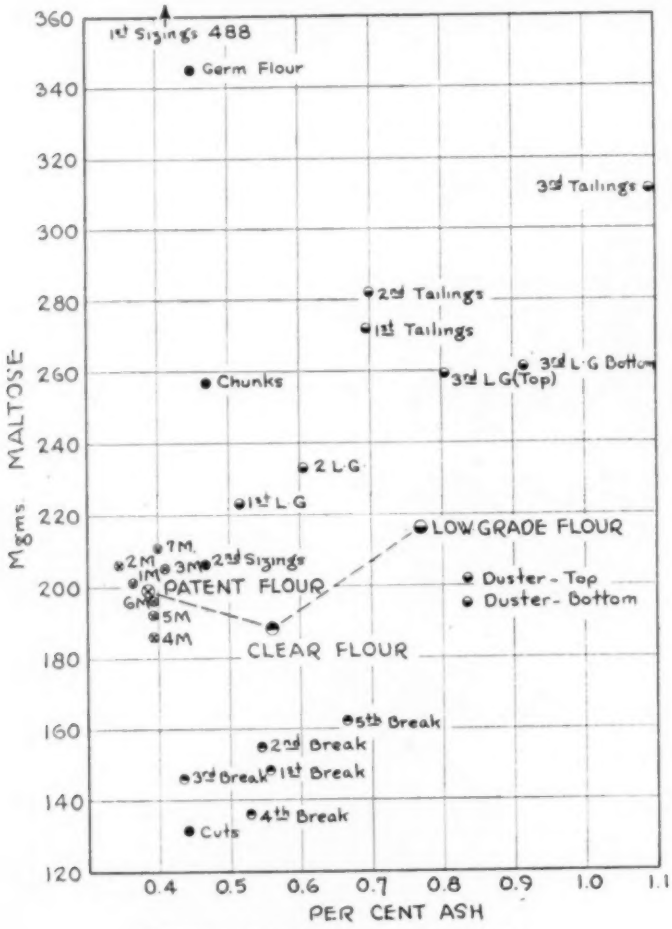

Figure 2. Correlation between maltose and ash.

be classified half-way between a break flour and a sizing flour. Most of the germ of the wheat berry is handled through the germ section and the first sizings in this particular mill. The " cuts " which are very low both in maltose and ether extract are a fine, very hard, sharp flour, which are dusted out of the middlings from the breaks. The graph plainly shows that the reason for the low maltose value on the clear flour is the

large percentage of break flour that goes into it. The tailings flour shows a high maltose value and also a high ether extract. This shows that there is a correlation between the ether extract and the maltose value, but that there are also other factors entering into it and that the parallelism is not absolute.

Figure 2 shows the correlation between the maltose and ash content. Here again we have the break flours falling in a rather close range, the middlings flours falling in another range, and the tailings flours showing a rather better correlation. Comparison of this figure with the one

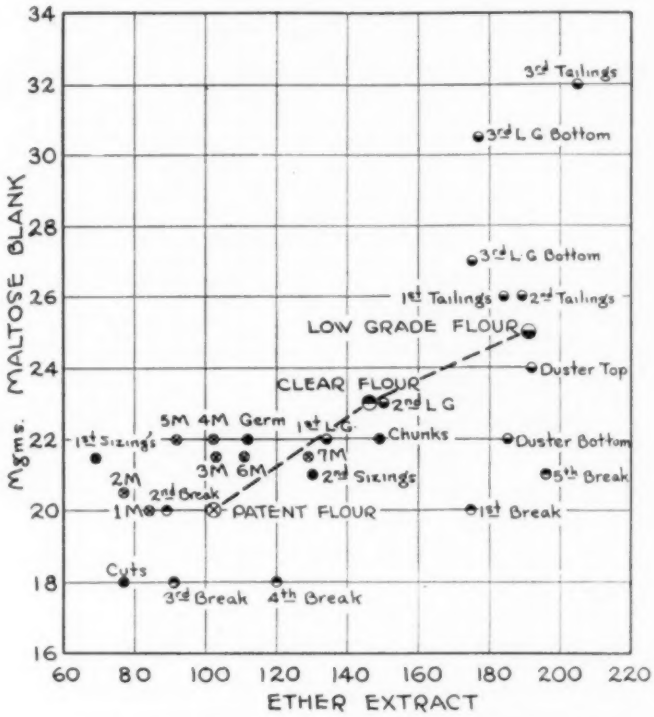

Figure 3. Correlation between maltose blank and ether extract.

between maltose and ether extract shows a decided similarity. This graph shows that while there is a fair correlation between maltose and ash, and should not be stated as a fact without qualifications, the influence of fineness of grinding and location from which the flour comes in the wheat berry is important.

In Figure 3, the correlation between the maltose blank and the ether extract shows the same relationship. The softer tailing flours yield a higher extract and show a larger maltose blank. This is particularly noticeable in the cuts flour as it is lowest in both respects. As before

mentioned, this flour is extremely hard and sharp and this characteristic may have something to do with the low results obtained. The finished patent flour, clear flour, and low grade flour show a fairly straight curve because the streams going to make up each one happened to average in this respect, and not because the flours always run parallel in their characteristics.

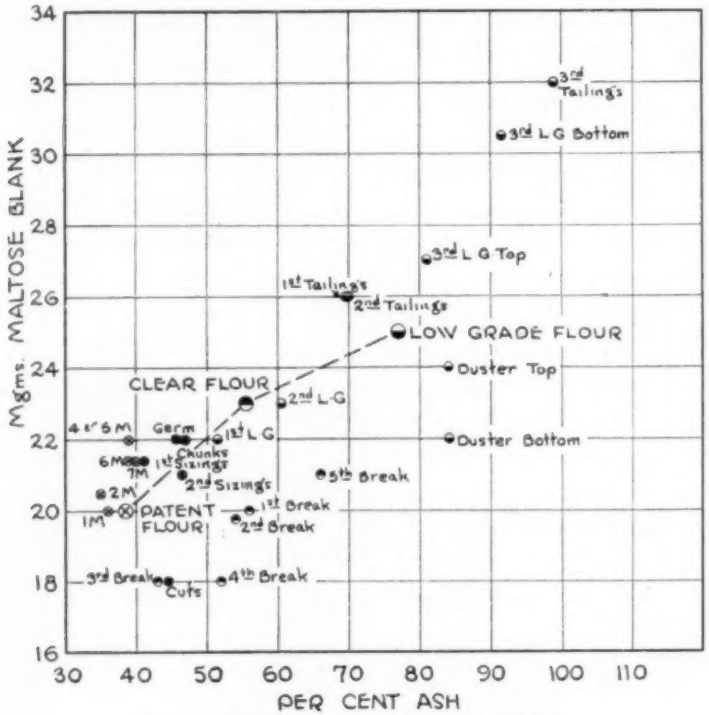

Figure 4. Correlation between ash and maltose blank.

In Figure 4 the correlation between the ash and the maltose blank is very similar in appearance to Figure 3.

Figure 5 shows that the correlation between the ash and ether extract is very good. It is interesting to note in this graph that the first sizing flour, which is one of the fine soft streams in the mill, and the cuts flour, which is the hardest and sharpest of all, lie closely together on this graph.

Examination of all these data and graphs would lead to the conclusion that the differences in diastatic activity and the response to malt treatment of these flour streams are due to a combination of (1) their degree of fineness, (2) the location within the wheat berry from which they come, and (3) the susceptibility of their starch, which is a function

to a certain extent of their fineness and also to the severity with which they have been ground before bolting.

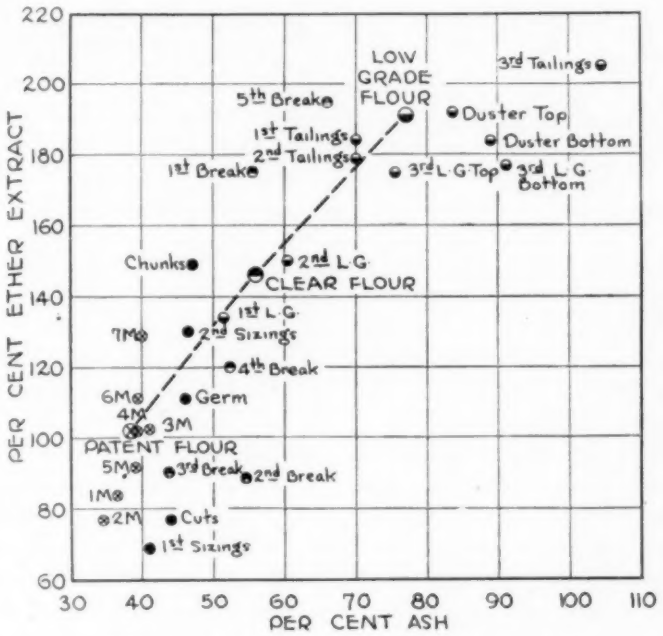

Figure 5. Correlation between ash and ether extract.

(C) Examination and Treatment of Stocks to Bring Out the Effect of Differences in Particle Size or Granulation

A sample of unbleached 100% flour was taken off the same 1800-barrel commercial mill. This mill was grinding at the time practically the same mill mix as shown in Table II. A sample of this flour was placed in a small rotomatic sifter clothed with 12-XXX, 13-XXX, and 14-XXX flour silks. It was allowed to bolt until examination indicated no more was passing through any of the sieves. This gave four separations of increasing degrees of fineness. No account was taken of the percentages of the different separations. The maltose was determined on these four separations before and after treatment with ¼ of 1% of malt flour. The results are shown in Table III.

The maltose values show a regular increase on the finer separations, and the finer stocks also show a greater susceptibility to malt treatment. This same experiment has been repeated a number of times with similar results. In this connection, it might be mentioned that two milling units, each of practically the same size, and flowed about the same except that one is clothed with finer silks, grinding identically the same mill

TABLE III

EFFECT OF PARTICLE SIZE

Separation (100% flour bolted)	Maltose	Maltose (+ ¼% malt flour)	Maltose increase
	Mg.[1]	*Mg.*[1]	*Mg.*
Scalp of 12-XXX	182	248	66
Through 12-XXX, scalp of 13-XXX	201	271	70
Through 13-XXX, scalp of 14-XXX	204	277	73
Through 14-XXX	211	296	84

[1] Milligrams of maltose from 10 g. of flour after 1 hour's diastasis at 30° C.

mix over a period of several months, showed this difference which granulation of the flour causes. The 100% on the mill clothed rather coarsely gave an average maltose result of 199 mg., whereas the one clothed with the finer silks showed an average of 231 mg. The reason for the finer clothing on one unit was the difficulty in keeping its flour reasonably free of specks.

The above conclusion, only as it bears on degree of fineness, was confirmed in the following manner. A very clean sample of purified middlings was obtained from the mill. This was divided into seven equal portions, and the different portions reduced to increasing degrees of fineness on the smooth rolls of an experimental mill. This was done by careful successive bolting and grindings until all the stock would pass through the sieve being used, and it was then thoroughly blended so as to be uniform. The diastatic activity was then determined on the six reduced samples, and also on the unreduced middlings. ¼ of 1% malt flour was added and the increase in maltose due to malt treatment noted. Results are shown in Table IV.

TABLE IV

PURIFIED MIDDLINGS REDUCTION

	Maltose	Maltose (+ ¼% malt flour)	Maltose increase
	Mg.[1]	*Mg.*[1]	*Mg.*
Purified middlings	72	113	41
Reground through 42-GG	76	117	41
Reground through 60-GG	95	136	41
Reground through 70-GG	108	150	42
Reground through 9-XXX	110	166	56
Reground through 11-XXXX	150	220	70
Reground through 13-XXX	178	260	81

[1] Milligrams of maltose from 10 g. of flour after 1 hour's diastasis at 30° C.

The stock through the 42-GG, the 60-GG, and the 70-GG, showed a fair increase in diastatic activity as the fineness increased, but the

response to malt treatment showed no increase. Stock through these grits gauzes is coarser than goes into the finished flour in an average flour mill. It appears that the starch granules were not very much affected, and the diastase was not enabled to attack it any more than that of the unground middlings. As the stock was ground finer, the increase both in the diastatic activity and the response to malt treatment increased rapidly. Previous experiments along this line have been made with a ball mill, but this shows that from a practical mill standpoint the finer the stock is ground the higher the diastatic activity and also the more susceptible the starch is to conversion by the diastase.

(*D*) Practicability of Experimental Milling Results in Estimating Diastatic Activity and Malt Treatment for the Purposes of Commercial Milling

In order to consider the possibility of the experimental mill being used to gain information regarding the proper malt treatment to give flour from any particular lot of wheat, the aid of five chemists was enlisted. A thoroughly mixed sample of Kansas hard winter wheat of 12.75% protein was divided into ten equal portions and two portions supplied to each of the collaborating chemists with the request that they mill each of the two samples supplied them on their experimental mills and send the flour to us. This was, in effect, a duplicate milling by each on the same wheat. The diastatic activity before and after treatment with ¼ of 1% of malt flour, was then run on these flour samples and the maltose increase noted. The results are shown in Table V.

TABLE V

Collaborative Experimental Milling

Chemist	Ash %	Maltose	Maltose (+ ¼% malt flour)	Maltose increase
		Mg.[1]	*Mg.*[1]	*Mg.*[1]
A	0.389	115	176	59
	.399	118	174	55
B	.385	130	187	57
	.394	130	185	55
C	.394	121	203	81
	.397	118	198	79
D	.453	136	198	62
	.440	130	192	62
E	.395	112	182	70
	.390	116	186	70

[1] Milligrams of maltose from 10 g. of flour after 1 hour's diastasis at 30° C.

Judging by the ash contents, all chemists excepting D obtained about the same degree of flour extraction. By slicking-up these five samples on a glass slide and looking through them at a source of light, it appeared that the diastatic activity on the untreated samples ran about parallel to their degree of fineness, except in the case of chemist C, whose flour appeared to be the finest. The next finest was chemist D. Although the results varied considerably between the different chemists, both on diastatic activity and increase from treatment, all showed a remarkably close check between duplicates in both respects. Unfortunately it was impossible to have commercially milled flours from these same chemists' mills as they would have provided interesting information. As we know from experience that a big mill grinding a uniform wheat mix varies very little from day to day in maltose value of the flour, it appears that it would be practical for any one of the five chemists to establish the relationship between the experimentally milled flour and the flour from the commercial unit, both as regards diastatic activity and response to malt flour treatment.

(*D1*) Experimental Mill Compared with Commercial Unit

On three successive days, samples of wheat were taken from the mill-mix going to a commercial unit and milled on an experimental mill, and the diastatic activity determined before and after malt treatment. Then at the time it was estimated this wheat mixture had reached the rolls of the commercial unit, a sample of the 95% flour was taken. The diastatic activity was run on this commercially milled flour before and after treatment with malt flour. The wheat mixture was identical on the three successive days, all coming from one mixture which had been mixed in the elevator and placed in the mill bins. Results are shown in Table VI.

TABLE VI

Experimental Mill Compared with Commercial Unit

	Experimental Mill			Commercial Mill		
	Maltose	Maltose (+ ¼% malt flour)	Maltose increase	Maltose	Maltose (+ ¼% malt flour)	Maltose increase
	Mg.[1]	*Mg.*[1]	*Mg.*[1]	*Mg.*[1]	*Mg.*[1]	*Mg.*[1]
	148	207	59	231	306	74
	152	210	58	228	309	81
	144	206	62	230	309	79
Average	148	208	59	229	308	78

[1] Milligrams of maltose from 10 g. of flour after 1 hour's diastasis at 30° C.

The relationship of the diastatic activity between the experimentally milled and the commercially milled flours was fairly uniform on the three days, as was the relationship between the two on maltose increase from malt treatment. Although it is not safe to draw a definite conclusion from these limited experiments, it appears that it would be

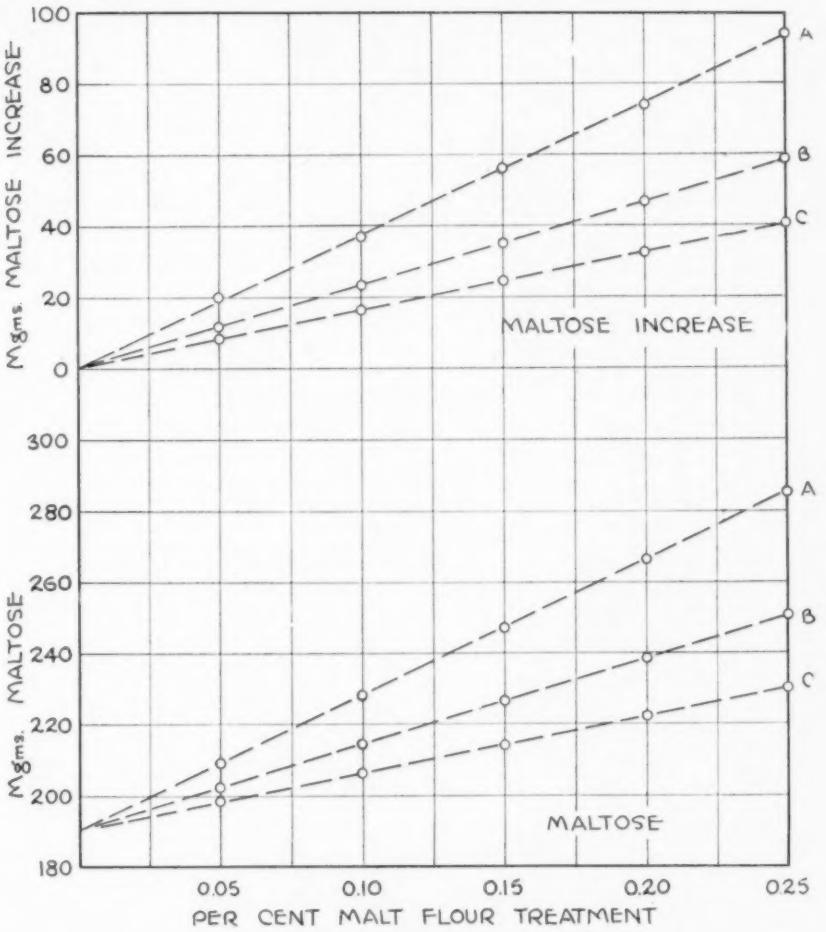

Figure 6. Comparison of the diastatic strength of three malt flours.

thoroughly practical to establish an average relationship between the experimental mill and the commercial unit both as regards initial diastatic activity and response to treatment. These results were secured on one type of wheat only, and differences in degree of fineness which might be secured working with other types of wheat might upset this relationship. It was our intention to go into this further by securing wheat

and flour samples from a number of mills grinding different types of wheat, but it was impossible to secure the samples and the data in time for presentation in this paper.

(*E*) Method for Comparing the Diastatic Strength of Different Malt Flours, etc.

A sample of untreated, unbleached, 80% bakers' patent flour made from Kansas hard wheat was treated with $^{5}/_{100}\%$, $^{1}/_{10}\%$, $^{15}/_{100}\%$, $^{2}/_{10}\%$, and $^{25}/_{100}\%$ of three different malted wheat flours. The diastatic activity was then determined on the untreated sample and on all the treated samples, and the increase from each treatment noted. These are shown in Figure 6. In the upper part of the graph the milligrams of maltose increase are plotted against the percentage treatment, and in the lower part, the milligrams of maltose as found are plotted against the percentage malt flour treatment.

Reference to either of the graphs brings out plainly the difference in the strength of the three malt flours. As the range of malt flour treatment generally employed in flour mills, on the average, is between $^{1}/_{10}$ of 1% and $^{25}/_{100}$ of 1%, it would be safe to judge the relative strengths of these three by averaging their gain between these points; or by tracing across on the graph between one curve and another, it is also possible to gain a very good idea of their relative power. This method may also be employed to determine the proper malt treatment to give any particular flour with any specific malt flour to obtain any desired maltose value figure. It is only necessary to treat the flour as shown, draw the graph, and then by reference to it, determine how much malt flour is necessary to secure the results desired. As a matter of fact, experience on many hundreds of samples has shown that if two points be established (0.1% and 0.25%) and a curve drawn as shown, results will be accurate enough for practical purposes.

Summary

Different grades of flour from different classes of wheat ground in different mills were tested for diastatic activity and their susceptibility to maltose conversion by treatment with malted wheat flour. The widely varying results obtained lead to the conclusion that both the initial maltose found and the maltose increase from treatment, by the methods employed, are a function of the variety or type of wheat ground, the location in the wheat berry from which the flour is derived, the way the stocks are classified in the different mills, and the fineness to which the stock is ground, and probably to the pressure exerted in grinding.

Experiments were conducted using the flour streams from an 1800-barrel mill grinding Kansas hard wheat. The flours were tested for ether extract, diastatic activity, and maltose increase when treated with malted wheat flour. Results indicate that both the diastatic activity and the response to treatment are a function of the location in the berry from which the stock is derived, the degree of fineness to which the stock is ground, and probably to the severity of pressure exerted in grinding. There is a fair correlation between ash and original diastatic activity, but other factors enter into it, and it is not absolute. Likewise, there is a fair correlation between the maltose and ether extract, but other factors, such as degree of fineness, also exert their influence. The correlation between the maltose blank and the ether extract is very similar to that between the maltose and the ether extract. The correlation between the ether extract and the ash is very good.

It was demonstrated that if an ordinary 100% flour was divided into portions according to degree of fineness, an increasingly higher diastatic activity was exhibited the finer the stock, and also that the finer stock is more susceptible to conversion by malted wheat flour.

It was also shown that if one stock is taken from a flour mill, and ground to varying degrees of fineness, the diastatic activity by this method increases as the stock becomes finer, and also the response to malted wheat flour treatment increases as the stock becomes finer.

A method is demonstrated whereby it is possible to judge the relative strength of malt flours by adding various percentages of the different malt flours to an untreated flour, and determining the maltose before and after treatment. Curves are then plotted and it is possible to compare the relative strength of the malt flours involved. This same method may be used to judge the necessary malt treatment to give an untreated flour in order to obtain any desired maltose content in the finished flour.

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THE THERMAL FRACTIONS OF GLUTEN PROTEINS AND THEIR RELATIONSHIP TO BAKING STRENGTH [1]

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Introduction

Early workers in the field of the wheat proteins considered a protein to be a definite chemical entity. Von Bibra (1861), Ritthausen (1872), Braconnot (1827), and Kossel (1900) pursued their attempts to isolate and identify vegetable proteins with this concept in mind.

Osborne (1909) classified the earlier work on the wheat proteins and by a series of brilliant researches accomplished more than all the earlier investigators combined. He postulated that these proteins were five in number—a proteose, a globulin, an albumin, gliadin, and glutenin, the latter two being the gluten proteins. The identity of these proteins as chemical entities was now generally accepted. Later, however, the homogeneity of glutenin and of gliadin came to be questioned, especially in the instance of glutenin, as well as the definition of wheat globulin. Csonka and Jones (1927), Blish (1926), and Blish and Sandstedt (1925, 1929, 1929a), Sinclair and Gortner (1933), Haugaard and Johnson (1930), and others have cast doubt upon the strict classification of the flour proteins into five chemical entities and have shown that overlapping solubilities due to various treatments, may cause erroneous estimation of the relative quantities of globulin, glutenin, and gliadin present. The likelihood of the existence of at least a third gluten protein has also been pointed out. This evidence appears to suggest a protein complex, which may be separated into various fractions, the relative proportions of these fractions varying according to the conditions to which the system is exposed.

Beccari (1745) first separated flour into two parts. This achievement marked the first isolation of crude gluten. Various other workers, as Parmetier, Taddei, Braconnot, *etc.*, carried on further investi-

[1] Condensed from a thesis presented to the Graduate School of the University of Minnesota by Rae H. Harris in partial fulfilment of the requirements for the degree of Doctor of Philosophy, November, 1935.

gations on gluten, such as drying and treatment with alcohol and saline solutions, and also endeavored to determine its composition and relation to other vegetable proteins. Ritthausen (1872) studied protein preparations and this work furnished the first broad foundation for further researches on the vegetable proteins. Osborne and Voorhees (1893) postulated that gluten contained two proteins, gliadin and glutenin, and this view was soon universally accepted. Osborne evolved a plan of separation of the gluten proteins by utilizing their varying solubilities in dilute salt solution and 70% ethyl alcohol and extended further than any one else in the field the current knowledge of the gluten proteins. Osborne in 1909 reviewed and consolidated the work of Ritthausen.

Snyder (1904) determined the gliadin concentration of 70% alcoholic flour extracts by means of the polariscope. Chamberlain (1906) and Tague (1925) showed that low results were obtained for glutenin due to removal of part of the gliadin as a portion of the soluble fraction when extracting with salt solution and alcohol. Olson (1913) extracted first with 65% alcohol, the alcohol was evaporated off and replaced by water, when presumably the total gliadin present settled out. Bailey and Blish (1915) isolated only 25% of the total gliadin by Olson's method. They confirmed the solubility of gliadin in dilute alcohol, as well as removal of some non-gliadin protein by various alcohol concentrations. Similar differences in alcohol solubilities were noted by Hoagland (1911) and Blish and Sandstedt (1929a).

Blish and Sandstedt (1925) noticed that H_2S was evolved when gluten was treated with concentrated alkali, the odor being less noticeable when weaker alkali was used, and proposed a method involving the dispersion of the flour in 0.1 N NaOH and methyl alcohol (65%). After filtering, the system was brought to a pH of 5.8 to 6.0 and the gliadin thrown down by centrifuging. Two entirely distinct methods for glutenin preparation were proposed by Blish, Abbott, and Platenius (1927). The first method consisted in extracting with ammonium hydroxide, then pouring the extract into 96% methyl alcohol. Glutenin precipitated out upon removing the ammonia, though 15% to 20% of the protein was not accounted for. The second procedure used barium hydroxide as extraction agent, followed by the addition of methyl alcohol to 75% concentration. The barium salts of glutenin precipitated out and were filtered off. Exposure to NaOH was eliminated in these methods, with resultant gain in concordance of results.

Glutenin, though formerly accepted as a chemical entity, has been subjected to grave doubts concerning its homogeneity as further studies have been carried out. Csonka and Jones (1927) appeared to succeed

in fractionating this protein into two components which they called α- and β-glutelins. These fractions showed evidence of a significant difference in nitrogen distribution, and thus apparently justified the authors' contention that they were chemically different. The alkali treatment may have caused the difference through cleavage of the protein complex. The α-glutelin precipitated at 0.018% to 0.020% saturation with ammonium sulphate, while a tenfold increase in concentration was required to throw down the β- fraction. Halton (1924) had previously suggested that glutenin consisted of two fractions, but was unable to repeat his original observations in this respect, and Gortner suggested that the apparent fractionation may have been due to racemization caused by the increased alkali concentration used by Halton.

Blish (1926) prepared chemically different glutenins from the same flour by varying the concentration of alkali used to disperse the residue subsequent to the alcoholic extraction. This led to further investigations of glutenin preparations by him in an attempt to obviate denaturation effects through the action of the solvents employed. A method was therefore proposed by him using dilute acetic acid instead of alkali as a dispersion medium (see Blish, 1930) thereby avoiding any irreversible alteration of the protein complex through the action of this reagent. He also opined that gluten contained at least two proteins in addition to gliadin.

Blish and Sandstedt (1929) isolated two samples of glutenin from two different flours using alkali concentrations, N/5 and N/60. The stronger alkali gave in each instance a product higher in amide and lower in basic nitrogen, which was explained by them as due to chemical change effected by the alkali. This led them to prepare glutenin by dispersion of gluten in dilute acetic acid, followed by neutralization with alkali and precipitation of the protein. The sample prepared in this manner resembled the original gluten very closely in appearance and properties. Blish and Sandstedt concluded that the new preparation was greater in molecular size and complexity than the glutenin formerly prepared by alkali treatment, which, they postulated, apparently splits off protein from the original glutenin.

The use of concentrated urea solutions according to the method of Cook and Alsberg (1931) was considered inadvisable by Blish on account of possible denaturation effects upon proteins, although urea probably produces less of this effect than most of the solvents used up to this time.

Larmour and Sallans (1932) compared five different methods for separating the gluten proteins, using one flour sample as the original material. They found no appreciable differences among the gliadins

prepared by the various procedures, but the glutenins varied in properties. The separation wherein 0.007 N acetic acid was used as dispersion medium was considered to be the most efficient on the basis of highest total nitrogen and lowest humin nitrogen.

Fractionation of Gluten Proteins

The first thermal fractionation of gluten protein was described by Dumas and Cahours (1843) who deposited a substance resembling gluten by cooling an alcoholic flour extract. Haugaard and Johnson (1930) fractionated gliadin prepared from wet gluten by treatment with 60% ethyl alcohol by volume. Upon progressively lowering the temperature gliadin fractions separated at 0° C. and — 11° C. The solution from the second isolation was concentrated under reduced pressure and a syrupy liquid finally obtained. Upon pouring this liquid into five times its volume of 1% NaCl solution a foam of gliadin was formed on the surface. This procedure was repeated using purified gliadin as the original material. The nitrogen content of these various fractions was not found to vary appreciably. Optical rotation determinations confirmed these findings. The precipitation temperature of the various gliadin fractions was found to rise with increasing increments of lithium chloride, reaching a maximum at 0.002 molar, and then decreasing with further salt additions. Gortner and Sinclair (1933) considered that the experimental evidence presented by Haugaard and Johnson (1930) did not warrant the conclusion that gliadin is a chemically heterogeneous substance.

Further fractionation studies indicated the possibility of separating out as many as 9 additional fractions from an original fraction by repeatedly dissolving and cooling the protein. When aliquot portions of these fractions were mixed together again, the final precipitation temperature coincided with the precipitation of the original plus 6° C. The different fractions were shown to be entirely reversible as the authors postulated; that is, gliadin exists as a reversible, fractionable, co-precipitation system.

Blish and Sandstedt (1933) discussed the separation of three distinct gluten proteins from dilute acetic acid-ethyl alcohol gluten dispersions on progressively lowering the temperature, provided that a small quantity of electrolyte was present. These three groups were designated as the glutenin group, the mesonin (to indicate the idea of intermediate properties) group, and the gliadin group. No sharp line of demarcation was postulated between these groups, but rather a gradual merging of one group into another. They considered the glutenin fraction to suffer the least dispersion in the solvents used, and to con-

sist of the largest particles. Gliadin is the most soluble of the three fractions in both the solvents employed, while mesonin was placed as being much less soluble in neutral alcohol, but highly so in dilute acetic acid, and would therefore be intermediate in properties between the other protein fractions. Using NaCl as the added electrolyte, they found glutenin to come down at 12° C., mesonin at about 2° C. and the remainder of the protein at — 12° C. Substituting K_2SO_4, the first precipitation occurred at about 19° C., and the other fractions had correspondingly higher temperatures than are necessary in the instance of NaCl. Moist crude gluten was used as the starting point as it was found that if flour itself was treated with alcohol or dilute acetic acid or a combination of the two the amount of glutenin dispersed was almost negligible. Gluten gave an opaque and unfilterable sol resembling a suspension more than a sol.

Sellers (1933) undertook to differentiate definitely the two gluten protein fractions, glutenin and mesonin, as prepared by the Blish method from moist crude gluten. The amino acids were separated according to the method of Brazier (1930) which consists in forming the copper salts of the individual amino acids, followed by separation of their metallic derivatives through solubility differences. Definite differences in nitrogen distribution were found between the two protein fractions isolated from 0.05 N acetic acid-ethyl alcohol (50%) medium containing a trace of K_2SO_4. Glutenin precipitated out at 18° C. to 20° C., mesonin at 12° C. to 14° C., while the gliadin remained in solution. The most significant difference obtained was in the amide nitrogen values, which were 12.344% for glutenin and 16.894% for mesonin. Sellers concluded that these two fractions are probably individual substances, and further postulated quite definitely that the glutelin of wheat flour is a heterogeneous material.

The peptization of gliadin by inorganic salt solutions was investigated by Gortner and Sinclair (*loc. cit.*). Two methods of preparation for the gliadin were employed, the original alcohol method of Osborne and the later Blish and Sandstedt acetic acid procedure. In successive treatments with molar KI solution a non-peptizable residue was always obtained. If this residue was purified by repeated precipitation from alcohol, further peptization by KI was possible. When treated in the same manner the reworked KI soluble fraction yielded an " insoluble " residue. No perceptible alteration was evident in the relative nitrogen distribution in the gliadin treated with the salt solutions as compared with the original, and Gortner thought a physical rather than a chemical change had occurred in the protein gel.

These authors discuss the " reversibly dissociable component sys-

tems" of Sørensen (1930) and point out that Sørensen's data are in agreement with the work done under Gortner's direction. Sørensen's theory is based upon the hypothesis that "soluble proteins consist of a series of complexes or components reversibly combined, which makes their constitution expressible by the ordinary formula $AxByCz\cdots$, A, B, and C marking complete complexes, mainly polypeptides, yet in some cases also containing other groups, for example, phosphorus ones, whereas the affixed indices x, y, and z, and so on, mark the amount to which the indicated complex is present in the entire component system." Linkages within each complex are through main valencies, but the complexes in the component system are knit together by the residual valencies each component is supposed to possess. These linkages are supposed to be comparatively slight and easily affected by alterations in the composition of the solution. Such alterations (salt content, hydrogen ion activity, alcohol content, temperature) may cause reversible dissociation of the components, with resultant interchange of components. When a component system more or less insoluble may be formed, this system will tend to be formed and precipitated. This theory is used to explain reversible fractionation of the proteins investigated, and disregards entirely the surface forces of colloid chemistry in aqueous protein solutions.

Gortner further states that the theory of Sørensen, Svedberg's "decomposition of protein molecules" and his own "protein peptization" really describe the same phenomena in different words. All three groups of workers have showed the possibility of fractionating protein material and later obtaining the original protein by specific technique.

Relationships between wheat proteins and baking strength

The relationships between various ratios of the wheat proteins and baking strength have been investigated without any definite agreement being reached among the various researchers. Snyder (1899) considered that a well-balanced gluten was composed of approximately 65% gliadin and 35% glutenin. Later (Snyder, 1904) this statement was modified to the gliadin content of 55% to 65% of the total protein in flour of good baking quality.

Shutt (1907) discussed protein composition in relation to baking strength and defined the relationships between determinations of nitrogen (protein, gliadin, and glutenin) and baking strength. No definite ratio was established, however.

Reichert (1902) thought that loaf volume was largely conditioned by the absolute quantity of gliadin present. The optimum value ranged between the limits 6% to 7.5%.

Blish (1916) studied the chemical constitution of the proteins of wheat flour and concluded that there was no essential difference in this respect between strong and weak flours. He also found the gliadin-glutenin ratio nearly constant, but great variability was evident in the percentages of the " soluble proteins."

Zinn (1923) correlated gliadin and total protein, and found significant correlation coefficients, ranging from $+ .9245 \pm .0181$ to $+ .7863 \pm .0243$. He concluded from these results that the gliadin varied with total protein.

Grewe and Bailey (1927) were unable to detect any significant variation in the ratio of glutenin to crude protein or of glutenin to the sum of glutenin and gliadin. They accordingly concluded that the glutenin-protein ratio was of little utility in distinguishing between various types of flour.

Sharp and Gortner (1923) found a constant ratio of gliadin to total protein in flours of widely different baking characteristics.

Lishkevich and Klyachina (1935) found no correlation between the various proteins in flour and baking strength. Fluctuations in protein content of wheat were found to be more apparent in relation to the geographic origin than to the variety. It was found that the gliadin-glutenin ratio was an index characteristic of groups of varieties, as well as of individual varieties. For durum wheat, the index was 0.765, for soft spring wheat 0.746, and for winter wheat 1.111.

Blish (1936) postulated that hydrolysis of gluten dispersions in dilute acetic acid at room temperature is mainly due to enzymatic action. This action can be effectively checked at ice temperature. This worker states that " mesonin " is a protein liberated upon disruption of a lipoid-protein complex, and this complex may play a rôle of fundamental importance in bread doughs. Alcohol apparently effects an irreversible splitting of the lecitho-protein.

Blish also states gluten may consist of either innumerable components differing " systematically and progressively," or of distinct groups, relatively few in number, which are thrown down as mixtures containing varying proportions of these groups. The second hypothesis is probably the correct view.

Experimental

Material and Methods

In view of the evidence pointing to the existence of at least three gluten protein fractions as separated by the thermal fractionation method of Blish and Sandstedt (*loc. cit.*), it seemed desirable to study the quantitative distribution of the three fractions in a series of widely di-

vergent flour types, and ascertain whether information relative to the agronomic or commercial value of these flours could be obtained.

Of the flours used in this study twenty were commercially milled, presumably from varieties of *T. vulgare,* and nine flours were experimentally milled from wheats other than *T. vulgare* as described in Table I. The *vulgare* wheat flours were of widely different baking characteristics, and included samples of soft wheat flour, biscuit flour, family and household flour, strong baker's patents, clears, millstream flour, *etc.*

After a number of preliminary studies the following protein fractionation method was adopted:

Method I.—50 g. of flour and sufficient tap water to make a stiff dough were thoroughly mixed to a dough in the Hobart-Swanson mixer. The dough was removed from the mixer, rounded up and placed in tap water at room temperature for approximately 30 minutes. The starch was then washed out under a small stream of running tepid water, care being taken to avoid any appreciable loss of gluten particles. This operation required about 10 minutes and an effort was made to hold the time to that limit. The gluten ball was pressed between the hands to remove as much of the water as possible, and weighed as wet gluten. 5-g. portions were immediately weighed out, cut into small pieces and placed in 200 cc. of 0.1 N acetic acid contained in 600 cc. beakers, covered with watch glasses, and allowed to stand 16 hours or longer at room temperature, with occasional stirring of the contents. At the end of this period, the gluten particles had dispersed, with the formation of a thick, turbid suspension, which was quite unfilterable. After thorough stirring, sufficient 95% ethyl alcohol was added to the dispersion with stirring to make a final concentration of 50% alcohol by volume, followed by 400 mg. of K_2SO_4. This salt was chosen because Blish had pointed out the convenient fractionation temperatures obtained by using it. It had also been used previously here for protein fractionations.

The beakers were next placed in a water bath at 18° C. to 20° C. for 48 hours to allow the "glutenin" to precipitate out. The supernatant liquid which was still quite turbid was decanted from the residual protein and the latter Kjeldahled, the glutenin fraction being calculated by multiplying by the factor 5.7. "Mesonin" was thrown down from the decantate upon standing 24 to 48 hours at 8° C. to 10° C. The liquid was centrifuged to remove suspended protein particles, and the precipitated material Kjeldahled. The protein was computed as mesonin as in the instance of the glutenin. The slightly opalescent liquid was aliquoted, the residual protein being determined as in the previous instances and recorded as gliadin.

The first or "glutenin" protein fraction obtained by this method was very small in amount, dirty grey in color, and did not vary greatly in quantity from sample to sample. The liquid did not appear to clear up appreciably following its precipitation. The second fraction which came down at 8° C. to 10° C. was much larger in amount, white in color, and had a slimy appearance. The supernatant liquid in this instance was much clearer than before, in many cases being practically water clear.

Method II.—A second procedure was evolved, differing from the first in only one particular, the K_2SO_4 (400 mg.) was added before the alcohol, stirred in with an electric stirrer for one minute, then the alcohol added. A preliminary investigation disclosed that increasing quantities of alcohol appeared to increase the quantity of glutenin precipitated, but a concentration of 50% by volume of ethyl alcohol was adhered to because of the smaller volume of liquid entailed. The use of larger quantities would doubtless cause less of the mesonin fraction to appear as such. The length of stirring time between addition of salt and dilution by alcohol was likewise investigated, one minute being found to give quite satisfactory results.

When using the second procedure, a copious precipitation of glutenin usually occurred immediately the dispersion was cooled to 18° C. to 20° C. This precipitate resembled the former "mesonin" fraction in appearance and consistency and was to all appearances the same protein. The supernatant liquid was now quite clear and could be almost entirely separated from the protein precipitate by decantation. The small portion remaining with the protein residue was removed by ordinary filtration without difficulty. Upon allowing the decantate to stand several days at 8° C. to 10° C., a second fine white precipitate was found to have settled upon the lower sides and bottom of the flask, leaving a water-clear supernatant liquid. This liquid was very easily quantitatively removed from the precipitated protein by decantation, as the mesonin adhered rather firmly to the glass and had to be removed with the aid of a policeman. This fraction was much less in quantity than the corresponding fraction obtained with Method I, and tended to form gradually as the temperature decreased. The clear liquid gradually became clouded, this turbidity growing more pronounced toward the lower portion of the flask as the protein commenced to settle out, but the cloudiness would disappear upon warming the sol. This effect was not noticeable when the first method of preparation was used, for here heat had no appreciable effect in clearing up the turbid suspension. This behaviour is in line with the observations of Blish, who remarked upon the irreversibility of the glutenin fraction, as contrasted with the rever-

sibility of the mesonin and gliadin fractions. Upon further cooling the mesonin decantate below 0° C., a deposit of gliadin was obtained. This precipitate was somewhat similar in appearance to the intermediate mesonin fraction, but had a finer, more vitreous character, very different from the first glutenin fraction.

The *T. vulgare* wheat flours were baked, using an "improver" formula similar to the formula already discussed in connection with the peptization studies with inorganic salt solutions. In the present instance 0.75% of diastatic malt (60° Lintner) was incorporated in the formula with 2 mg. $KBrO_3$ in addition to the usual baking ingredients. A mixing time of two minutes in the Hobart-Swanson mixer was considered to be sufficiently long without introducing any danger of overmixing in view of the somewhat drastic formula employed. A baking score was calculated for these loaves according to the following formula: $0.1 \times$ (loaf volume -200) $+$ texture score $+ 2 \times$ the grain score. A standard loaf was baked each day from a good quality patent flour and this loaf was arbitrarily assigned a value of 10 for grain and texture characteristics. This baking score is an attempt to resolve into one value the loaf factors which indicate flour strength. The approximate volume of the moulded loaf is 200 cc. and therefore the volume of the baked loaf -200 cc. is the expansion in proofing and baking. Grain is emphasized as being more important than texture. A color score is not included, as it is distinct from strength as the latter term is employed here.

Discussion

In Table I the data are divided in two parts corresponding to the genetic classification of the original wheats. Within these divisions the flours are arranged in order of increasing crude protein, as this constituent is generally considered to be an important factor in rating flour for baking purposes and also tends to bring out any marked trend in the data upon visual inspection. Ash content, percent of wet crude gluten, loaf volume, and strength score of the *vulgare* flours are also recorded, while the non-*vulgare* flours were not subjected to baking tests. A range of 7.4% to 17.5% in crude protein was encountered in the *vulgare* wheats, and a range from 9.3% to 16.6% for the non-*vulgare* flours. An increase in wet crude gluten and loaf volume is evident in proceeding from the lower to the higher protein flours. This increase is not so apparent in the strength score. The *vulgare* wheat flour tends to yield higher wet gluten values than the non-*vulgare* flours of corresponding crude protein content. In several cases, as in Nos. 26 and 28, this may be due to some extent to the greater difficulty encountered in washing out the starch. The gluten in these flours disintegrated under

the action of water and it was found necessary to add 4% of K_2SO_4 to the water used to "dough up" these samples before washing. In addition, these doughs were made with less than the usual quantity of water.

TABLE I

DESCRIPTION AND DATA ON FLOURS USED IN THIS STUDY. (13.5 moisture basis)

Sample No.	Description	Ash	Crude protein N × 5.7	Wet crude gluten	Loaf volume	Strength score
		%	%	%	Cc.	
	Commercially milled *vulgare* wheat flours.					
1.	Cake flour milled from Indiana wheat	0.39	7.4	26.8	—	—
2.	Cake flour, Pacific Coast	.34	8.0	27.6	380	43
3.	Semi-hard Palouse Federation	.42	8.6	26.9	447	49
4.	Cracker pastry, Illinois	.41	8.7	31.8	434	53
5.	German clear, Germany	.50	8.9	29.3	470	43
6.	Minnesota cake flour	.26	9.4	29.2	392	45
7.	Low protein flour, Illinois	.39	10.4	38.3	528	62
8.	Biscuit flour, Pacific Coast	.42	11.1	34.8	508	57
9.	Minnesota family flour	.29	11.5	40.0	490	62
10.	Minturki clear, Univ. Farm, Minn.	.57	12.1	41.7	421	28
11.	Minnesota State Mill winter wheat straight	.41	12.5	40.3	457	43
12.	Big Bend high protein Bart	.37	12.5	44.0	568	66
13.	Minnesota strong baker's flour	.47	12.6	44.5	510	61
14.	First clear, Kansas	.56	12.6	43.0	530	60
15.	Marquis wheat flour, Crookston, Minn.	.39	12.7	44.5	588	66
16.	Second middlings flour, State Mill (Minn.)	.35	13.0	41.0	500	54
17.	Marquis wheat flour, Fort Benton, Mont.	.45	14.0	47.7	563	60
18.	Second clear, Kansas	.89	14.6	52.5	583	56
19.	Marquis wheat flour, Fargo, N. Dak.	.41	16.2	54.6	620	68
20.	Third break, State Mill (Minn.)	.65	17.5	63.7	618	64
	Experimentally milled flours from other than *vulgare* wheats.					
21.	Little Club, Pendleton, Ore. (*T. vulgare* x *T. compactum*)	0.50	9.3	29.1		
22.	Spelt, Univ. Farm, Minn. (*T. spelta*)	.62	11.0	37.6		
23.	Mindum, Univ. Farm, Minn. (*T. durum*)	.68	12.5	33.5		
24.	Poulard, Pullman, Wash. (*T. turgidum*)	.77	13.5	45.4		
25.	Polish, Bozeman, Mont. (*T. polonicum*)	.88	13.9	37.2		
26.	Einkorn (*T. monococcum*)	.60	14.4	36.3		
27.	Poulard, Univ. Farm, Minn. (*T. turgidum*)	.99	14.8	46.0		
28.	Yaroslav, Langdon, N. Dak. (*T. emmer*)	1.55	15.6	43.0		
29.	Polish, Pullman, Wash. (*T. polonicum*)	.75	16.6	47.5		

The majority of the glutens washed from the low protein *vulgare* wheat flours felt smooth and silky, and were white in color with fine texture. These characteristics were especially noticeable for No. 6, while No. 5 was probably the worst in these respects. The four highest protein flours, Nos. 17, 18, 19, and 20, had strong glutens while the flours intermediate between the high and low groups produced glutens generally lacking in the fine, silky texture of the low protein, cake flour class but of good elasticity. These gluten characteristics corresponded to the "feel" and appearance of the doughs made from these flours for baking purposes. The glutens of the non-*vulgare* wheats varied greatly in character, but were on the whole much inferior in physical properties

to the bread wheats. Flours milled from einkorn and emmer were rated the poorest in gluten quality, while the poulard (*T. turgidum*) appeared to be the best.

In Table II are recorded the weights in grams of the three protein fractions obtained by fractionating 5 g. of wet crude gluten by the procedures outlined. The sum of the three fractions in each preparation is also recorded. An examination of the data presented in this table reveals no marked trends in the protein fractions with increasing

TABLE II

PROTEIN DISTRIBUTION IN GRAMS AND AS PERCENTAGES OF TOTAL OF THE THREE PROTEINS OBTAINED BY TWO METHODS OF FRACTIONATING 5 GRAMS OF WET CRUDE GLUTEN [1]

Sample No.	Glutenin *a*		Mesonin *a*		Gliadin *a*		Total	Glutenin *b*		Mesonin *b*		Gliadin *b*		Total
	g.	%	g.	%	g.	%		g.	%	g.	%	g.	%	
1.	0.06	5.1	0.51	40.0	0.70	54.9	1.27							
2.	.07	6.2	.48	42.4	.59	51.4	1.14	0.42	37.5	0.16	13.9	0.55	48.6	1.13
3.	.06	4.7	.50	39.1	.71	56.1	1.27	.35	29.8	.19	16.0	.64	54.2	1.18
4.								.45	39.3	.15	13.1	.54	47.6	1.14
5.	.08	6.2	.46	36.8	.71	57.0	1.25	.36	30.5	.17	14.9	.64	54.6	1.17
6.	.05	3.8	.53	43.2	.65	53.0	1.23	.49	42.6	.14	12.0	.53	45.4	1.16
7.								.41	35.1	.16	14.1	.59	50.8	1.16
8.	.04	3.4	.45	36.8	.73	59.8	1.22	.32	29.1	.18	16.3	.60	54.6	1.10
9.	.06	4.8	.43	35.2	.73	60.0	1.22	.40	33.8	.19	15.7	.60	50.5	1.19
10.	.06	4.8	.43	32.6	.83	62.6	1.32	.40	33.7	.13	11.2	.65	55.1	1.18
11.	.08	6.4	.45	35.9	.73	57.7	1.26	.41	32.7	.20	15.9	.64	51.4	1.25
12.	.05	4.6	.47	41.3	.61	54.1	1.13	.35	31.1	.17	14.7	.62	54.2	1.14
13.	.10	8.0	.42	34.1	.71	57.9	1.23	.46	37.7	.13	10.8	.63	51.5	1.22
14.	.05	3.6	.37	31.1	.78	63.3	1.20	.42	37.5	.16	13.8	.55	48.7	1.12
15.	.06	4.3	.47	34.8	.82	60.9	1.35	.44	35.1	.16	12.4	.66	52.5	1.26
16.	.08	5.9	.57	43.0	.68	51.1	1.33	.48	37.1	.18	13.7	.64	49.2	1.30
17.	.07	5.7	.39	29.7	.85	64.6	1.31	.46	35.8	.12	9.7	.70	54.5	1.28
18.	.05	3.2	.28	19.5	1.11	77.3	1.44	.40	31.8	.18	14.0	.68	54.2	1.26
19.	.20	13.7	.57	39.1	.68	47.2	1.45	.45	35.9	.12	9.2	.69	54.9	1.26
20.	.06	4.4	.42	32.0	.83	63.6	1.31	.44	33.9	.16	12.1	.69	54.0	1.29
21.	.03	2.5	.40	34.2	.73	63.3	1.16	.33	32.1	.14	13.6	.55	54.3	1.02
22.	.04	3.4	.50	42.0	.65	54.6	1.19	.38	32.3	.20	17.3	.59	50.4	1.17
23.	.08	6.7	.43	34.3	.75	59.0	1.26	.42	33.3	.15	11.5	.69	55.2	1.26
24.	.05	4.6	.21	19.0	.85	76.4	1.11	.35	31.7	.12	10.7	.64	57.6	1.11
25.	.09	6.6	.48	33.7	.84	59.7	1.41	.46	33.7	.11	8.1	.80	58.2	1.37
26.	.03	2.8	.26	21.3	.94	75.9	1.23	.28	25.6	.14	12.6	.67	61.8	1.09
27.	.03	2.8	.26	22.6	.87	74.6	1.16	.28	26.6	.12	11.1	.66	62.3	1.06
28.	.07	8.5	.20	23.4	.58	68.1	0.85	.36	28.0	.16	12.2	.77	59.8	1.29
29.	.05	4.0	.09	6.8	1.21	89.2	1.35	.39	33.0	.09	7.5	.70	59.5	1.18

[1] Due to the fact that the protein fractions separated by the two methods of isolation did not appear to be identical the fractions isolated by Method I are designated as glutenin *a*, mesonin *a*, *etc.*, those isolated by the second method as glutenin *b* and so on.

crude flour protein for fractionation Method I. A somewhat different picture is shown by the data obtained by Method II, where a decided trend toward an increase in the gliadin fraction is evident with increasing crude protein. The quantity of glutenin isolated by Method I is

very small, while the mesonin fraction is much larger than would be expected from a knowledge of the literature related to gluten protein fractionation. Much larger quantities of protein are isolated as glutenin by the second method, with less present in the other two fractions.

The protein fractions calculated as percent of total of the three proteins are also shown in Table II. These data handled in this manner present more clearly the relative quantities of protein isolated by the

TABLE III

PROTEIN FRACTIONS BY METHOD I CALCULATED AS PERCENT OF FLOUR

Sample No.	Glutenin *a*	Mesonin *a*	Gliadin *a*	Total crude gluten protein	Crude protein − Total crude gluten protein
	%	%	%	%	%
1.	0.35	2.74	3.76	6.85	0.55
2.	.39	2.67	3.23	6.29	1.71
3.	.32	2.67	3.83	6.82	1.78
5.	.46	2.68	4.15	7.29	1.61
6.	.27	3.10	3.79	7.16	2.24
8.	.30	3.15	5.12	8.57	2.53
9.	.47	3.43	5.86	9.76	1.74
10.	.52	3.60	6.90	11.02	1.08
11.	.65	3.65	5.87	10.17	2.33
12.	.45	4.10	5.38	9.93	2.57
13.	.88	3.73	6.33	10.94	1.66
14.	.37	3.18	6.67	10.22	2.38
15.	.52	4.18	7.31	12.01	0.69
16.	.64	4.70	5.59	10.93	2.07
17.	.72	3.73	8.13	12.58	1.42
18.	.49	2.95	11.66	15.10	−0.50
19.	2.16	6.19	7.47	15.82	0.38
20.	.73	5.34	10.64	16.71	0.79
21.	.17	2.30	4.26	6.73	2.57
22.	.30	3.76	4.88	8.94	2.06
23.	.57	2.91	5.00	8.48	4.02
24.	.47	1.93	7.76	10.16	3.34
25.	.69	3.55	6.28	10.52	3.38
26.	.25	1.92	6.83	9.00	5.40
27.	.30	2.42	7.97	10.69	4.11
28.	.62	1.71	4.97	7.30	7.50
29.	.51	0.88	11.50	12.89	3.71

two methods. A lower percentage of glutenin is separated by Method I, but a higher mesonin and gliadin percentage, than the corresponding values obtained by Method II. No definite trends are evident when the percentages of protein in a fraction isolated by one method are compared with the corresponding fraction values isolated by the other method.

In Table III are shown the protein fractions obtained by Method I calculated as percent of flour, with total crude gluten protein (sum of

the fractions) percentage present. Non-gluten protein values (crude flour protein—sum of the fractions) were also calculated and recorded. Corresponding data yielded by Method II are presented in Table IV, with the gliadin *b*/glutenin *b* and gliadin *b*/mesonin *b* ratios. From the data contained in these tables it appears that the genus *vulgare* has more crude protein capable of forming crude gluten than in the instance of

TABLE IV

PROTEIN FRACTIONS BY METHOD II CALCULATED AS PERCENT OF FLOUR. GLIADIN/GLUTENIN AND GLIADIN/MESONIN RATIOS

Sample No.	Glutenin *b*	Mesonin *b*	Gliadin *b*	Total crude gluten protein	Crude protein − Total crude gluten protein	Gliadin *b* / Glutenin *b*	Gliadin *b* / Mesonin *b*
	%	%	%	%	%		
2.	2.34	0.87	3.04	6.25	1.75	1.30	3.50
3.	1.90	1.02	3.44	6.36	2.24	1.81	3.38
4.	2.83	0.95	3.43	7.21	1.49	1.21	3.62
5.	2.08	1.02	3.73	6.83	2.07	1.79	3.65
6.	2.89	0.82	3.08	6.79	2.63	1.06	3.77
7.	3.15	1.26	4.56	8.97	1.43	1.45	3.61
8.	2.21	1.24	4.16	7.61	3.49	1.88	3.34
9.	3.24	1.50	4.84	9.58	1.92	1.50	3.23
10.	3.30	1.10	5.41	9.81	2.29	1.64	4.93
11.	3.29	1.61	5.18	10.08	2.42	1.57	3.22
12.	3.12	1.48	5.42	10.02	2.48	1.74	3.67
13.	4.09	1.18	5.58	10.85	1.75	1.36	4.74
14.	3.64	1.34	4.72	9.70	2.90	1.30	3.52
15.	3.95	1.39	5.90	11.24	1.46	1.49	4.23
16.	3.98	1.47	5.27	10.72	2.28	1.33	3.57
17.	4.36	1.18	6.64	12.18	1.82	1.52	5.62
18.	4.21	1.85	7.18	13.24	1.36	1.71	3.88
19.	4.92	1.27	7.53	13.72	2.48	1.53	5.95
20.	5.55	1.98	8.84	16.37	1.13	1.59	4.45
21.	1.90	0.80	3.20	5.90	3.40	1.69	4.00
22.	2.83	1.52	4.41	8.76	2.24	1.52	2.95
23.	2.82	0.98	4.67	8.47	4.03	1.65	4.77
24.	3.22	1.09	5.86	10.17	3.33	1.82	5.35
25.	3.44	0.83	5.94	10.21	3.69	1.73	7.13
26.	2.01	0.99	4.85	7.85	6.55	2.41	4.91
27.	2.61	1.09	6.12	9.82	4.98	2.35	5.62
28.	3.10	1.35	6.63	11.08	4.52	2.14	4.92
29.	3.67	0.84	6.62	11.13	5.47	1.80	7.86

the other wheats. The conclusion applies to the values obtained by both methods of protein fractionation.

The mean percentages of the three protein fractions isolated by the two methods are shown in Table V. These values are classified separately for the two wheat groups to bring out possible differences in the protein distribution between these wheats.

TABLE V

COMPARISON OF THE MEAN PERCENT OF GLUTEN FRACTIONS IN *Vulgare* AND NON-*vulgare* WHEAT FLOURS

Wheat Group	Method I			Method II		
	Glutenin *a*	Mesonin *a*	Gliadin *a*	Glutenin *b*	Mesonin *b*	Gliadin *b*
	%	%	%	%	%	%
Vulgare	5.5	35.9	58.6	34.7	13.4	51.9
Non-vulgare	4.6	26.4	69.0	30.7	11.6	57.7

From the data presented in this table it is evident that the glutenin and mesonin fractions are higher in the group of *T. vulgare* flours than in the other genetic species. The gliadin percentages, however, are higher for the non-*vulgare* wheat varieties. It appears from the values shown that *vulgare* wheat varieties have more of the gluten proteins present in the potential form of glutenin and mesonin and less as gliadin in comparison with the wheats classified as other than *vulgare* types. The difference in percent of gluten protein isolated in the various fractions by the two methods is brought out strikingly by the data presented in this table.

A study of the correlation coefficients presented in Table VI reveals significant correlations between the crude protein content and the quantity of gliadin isolated by both methods, this relationship being higher in the case of Method II. This is true for the entire series of 29 as well as for the bread wheat flours. The glutenin and mesonin fractions, on the other hand, do not appear to have a relationship of any great importance with crude protein content.

Loaf volume is significantly correlated with crude protein, and with the quantity of gliadin present in the flours of the *vulgare* wheats. The same conclusion was reached by Zinn, and Sharp and Gortner. Glutenin and mesonin are not significantly related to loaf volume when these proteins are calculated in grams per 5 g. of wet crude gluten. When the three protein fractions were computed as percent of flour, however, all showed significant correlations with loaf volume. This is in agreement with the conclusions of Grewe and Bailey, and Blish.

Strength score, as calculated, does not have a relationship of any great importance to loaf volume or gliadin content but is significantly correlated with crude flour protein. The lack of relationship of loaf volume with strength score is probably due to the inferior grain and texture of some of the larger loaves.

Wet crude gluten was related to loaf volume and crude flour pro-

TABLE VI

CORRELATION COEFFICIENTS COMPUTED FROM THE FRACTIONATION AND BAKING DATA

Variables correlated		Method I			Method II					
		Number in sample	r_{xy}	P [1]	Number in sample	r_{xy}	P	Number in sample	r_{xy}	P
x	y									
Crude protein	Gliadin	27	+.3951	.0409	28	+.7265	<.0001			
"	Gliadin	18	+.4872	.0394	19	+.7214	.0003			
"	Mesonin	18	−.2897	.2340	19	−.2792	.2519			
"	Glutenin	18	+.3282	.1872	19	+.2518	.3039			
Loaf volume	Gliadin				19	+.6677	.0013			
"	Mesonin				19	−.1653	.5048			
"	Glutenin				19	+.0242	>.5000			
"	Gliadin (% of flour)				19	+.8427	<.0001			
"	Mesonin (% of flour)				19	+.6993	.0005			
"	Glutenin (% of flour)				19	+.7408	.0001			
"	Gliadin/Mesonin				19	+.4802	.0367			
"	Gliadin/Glutenin				19	+.3342	.1647			
Strength score	Gliadin				19	+.2317	.3458			
"	Gliadin/Mesonin				19	+.2345	.3498			
"	Gliadin/Glutenin				19	+.0086	>.5000			
Crude protein	Wet crude gluten							19	+.8805	<.0001
"	Loaf volume							19	+.8285	.0001
"	Strength score							19	+.5102	.0245
Loaf volume	Strength score							19	+.2516	.3044
"	Wet crude gluten							19	+.8461	<.0001

[1] P = the probability of the observed correlation coefficient arising from uncorrelated material through errors of random sampling.

tein, as would be expected from a general conception of the relationship between these variables.

Summary and Conclusions

Wet crude gluten was washed from 20 samples of *vulgare* wheat flour and 9 samples of flour milled from various non-*vulgare* wheats. These glutens were then fractionated into three distinct components by dispersion in 0.1 N acetic acid, addition of K_2SO_4, of ethyl alcohol to 50% concentration by volume, then progressively lowering the temperature when glutenin came down at 18° C. to 20° C., mesonin at 8° C. to 10° C. Gliadin was determined upon an aliquot of the residual solution. These fractions were quite distinct in appearance and physical properties although the mesonin appeared to be intermediate between the other two. Whether the electrolyte was added before or after the alcohol influenced the relative quantities of glutenin and mesonin isolated. Gliadin was also affected to a lesser degree. Addition of the K_2SO_4 to the acetic acid dispersion before the alcohol was added (Method II) appeared to yield more satisfactory results in point of clear separation of the respective gluten protein fractions. These results were also in better agreement with the values for glutenin and mesonin previously obtained by other workers.

The quantity of gliadin obtained by fractionating 5 g. of wet crude gluten washed from the various flours was significantly and positively correlated with crude protein and loaf volume. This was not true in the instance of the other two fractions. When the three proteins were computed as percent of flour, positive significant correlations with loaf volume were evident in every case. This was probably due to the influence of crude protein.

No further information relative to the loaf volume of the flours appeared to be gained by thermal fractionation of the gluten proteins in addition to that obtained from a knowledge of crude protein. The gliadin *b*/glutenin *b* or gliadin *b*/mesonin *b* ratios were not correlated with loaf volume.

Vulgare wheat flours appeared to have more of the crude protein present capable of forming crude gluten than is the case with other species. *Vulgare* wheats also appear to have more of the glutenin and mesonin fractions present in the crude protein, and less of gliadin, than do wheats of non-*vulgare* varieties.

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A STUDY OF SOME PHYSICAL PROPERTIES OF FLOUR DOUGHS IN RELATION TO THEIR BREAD-MAKING QUALITIES

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Introduction

Wheaten flour is manufactured primarily for bread-making purposes and in consequence the most commonly used test of flour quality is the baking test. While, however, this is sound in principle, satisfactory practical methods of test baking are difficult to attain and much work has been done both in America and in Europe in attempts to standardise the baking test. An appreciation of the difficulties of this test has led to the introduction of other methods of testing flour, but while many of these have found favour in limited circles, it is true to say that there is at the moment no test available which meets with universal acceptance.

As our knowledge has grown, it has become increasingly obvious that flour quality is determined by a number of separate physical properties of flour and that all tests so far introduced in substitution for the baking test either measure one of these properties only, in which case the story is incomplete, or else they provide a composite picture of the effects of all the factors acting together, in which case the magnitude and the mutual relationships of the separate properties cannot be assessed and the picture obtained is often misleading and sometimes incorrect.

The present position is therefore far from satisfactory and the need for the isolation and individual examination of those flour properties which contribute to baking quality is urgent.

It is now generally realised that the production of satisfactory bread from any given flour depends not only on the intrinsic quality of the flour itself but also on the production of sufficient gas during the

[1] By mutual agreement the authors' names are given in alphabetical order, and no seniority is implied.

various stages of the fermentation of the dough. A realisation of this was first due to Wood (1907), the importance of whose work was emphasised over a period of years by Humphries (1909) and his collaborators. It has been again emphasised by Fisher and Halton in 1929, and subsequent workers (see Jørgensen, 1931).

The study of bread-making quality, apart from the gassing problem, has been followed along many lines, and as a result of the failure of the earlier work to connect quality with the chemical changes that go on in a fermenting dough attention has been focussed during recent years on the physical condition of dough. Investigations into the physical properties of dough have been, however, of an empirical nature and, apart from the work of Kosutány (1907), no attempt has been made to separate and individually measure definite physical constants until the work of Schofield and Scott Blair (1932, 1933, 1933a).

When a yeasted dough ferments, chemical changes occur which result in the production of carbon dioxide. Under pressure of this gas the dough is inflated and undergoes physical changes as a result of the change in shape. The physical changes that occur are dependent on the plastic and elastic nature of dough.

Dough is neither a true solid nor a true fluid but possesses properties associated with both states of matter. Solids can be differentiated from liquids by the fact that while the former have shape the latter have no shape of their own but take that of the vessels containing them. The shape of solids can be deformed by force but, due to their elastic nature, they will regain their original shapes when the deforming force is removed provided the deformation does not exceed a certain magnitude. If a piece of dough is moulded into a ball and then momentarily squeezed between the fingers it "springs back" on release thus behaving like an elastic solid. On the other hand, if the ball is left to stand it does not keep its original shape but flattens out to some extent. In this way it behaves like a liquid showing the property of flow.

The reason why dough combines the elastic properties of a solid with the viscous properties of a liquid is due to its peculiar internal structure. Flour dough contains protein chains which behave like coiled springs and are responsible for its elastic behaviour. These protein chains are not, however, linked to each other at all points with equal strength. When the dough is extended some of the linkages break almost at once, causing deformations which cannot be recovered (flow), while others maintain the rigid structure of the dough. All these adjustments in the protein network have to take place in the presence of a starch-water mixture which, although primarily fluid,

also possesses some rigid properties, thus complicating the situation, and making impossible the complete relaxation of even those protein units which are capable of truly elastic recovery.[2]

In view of the possible dependence of baking quality on the physical properties of dough, the present study was started with the object of obtaining measurements of these properties on doughs made from a variety of flours of different characteristics. It was thought that this would lead to a better insight into the mechanism of dough behaviour in the bakehouse and that eventually it might be possible to express baking quality in figures obtained from these measurements.

Elastic and Plastic Properties of Dough

VISCOSITY AND SHEAR MODULUS.—The extent to which an elastic solid deforms under force is determined by its elastic moduli. Young's modulus or the modulus of stretch is given by the ratio

$$\frac{\text{Loading force per unit cross-section}}{\text{Stretch per unit length}}.$$

The loading force per unit cross-section is known as the tensile stress and the stretch per unit length as the tensile strain.

The shear modulus (n) of an elastic solid is equal to the ratio $\frac{\text{shearing stress}}{\text{shearing strain}}$, and for dough the shear modulus is equal to one-third Young's modulus since the shearing stress is equal to one-third of the tensile stress and the shear and tensile strains are numerically equal.[3]

If a perfectly elastic solid is loaded and then unloaded the stretch and recovery are equal, but for dough or any other similarly plastic material the stretch is partly elastic (recoverable) and partly plastic (non-recoverable). To measure the shear modulus of dough it is therefore necessary to load a sample with a known force per unit area and measure the elastic recovery per unit length when the force is removed.

If, therefore, a dough cylinder of original length l_1 cm. is subjected to a shearing stress of S dynes/cm.^{2} so that it stretches to a length l_2 cm., at which point the stress is removed and the cylinder contracts to length l_3 cm., the elastic recovery[4] or strain is $\frac{l_2 - l_3}{l_3}$ cm. per cm. and

$$n = S \Big/ \frac{l_2 - l_3}{l_3}.$$

The ease with which a liquid flows is determined by the internal

[2] See Schofield and Scott Blair (1936).

[3] This is because dough does not change its volume appreciably on stretching. For a fuller explanation see Schofield and Scott Blair (1932).

[4] This is only strictly true for small extensions, see Schofield and Scott Blair (1933).

friction between the planes of the liquid. The coefficient of internal friction or viscosity (η) is given by the relationship

$$\eta = \frac{\text{shearing stress per cm.}^2}{\text{rate of flow}}.$$

If a dough cylinder of length l_1 is subjected to a shearing stress of S dynes/cm.^{2} for t seconds, so that it increases to length l_2, and then the stress is removed so that the length contracts to l_3, the permanent non-elastic change in length is $\frac{l_3 - l_1}{l_1}$ cm. per cm. and the rate of viscous flow therefore $\frac{l_3 - l_1}{l_1} \Big/ t$. The viscosity of the dough is therefore given by $\eta = S \times t \Big/ \frac{l_3 - l_1}{l_1}$. In practice, the viscosity of a dough never stays constant during an extension, so that $\frac{l_3 - l_1}{l_1} \Big/ t$ must be regarded as a mean rate of flow, and η as a mean viscosity.

"Elastic After-Effect" and "Hysteresis."—If a piece of dough is stretched between the hands and then released it will be noticed that the contraction in length is at first rapid but afterwards continues slowly for some minutes. This slow contraction is known as "elastic after-effect" and is due to inhibition of elastic recovery due to viscous resistances in the dough. If experiments are done at such a rate as to prevent complete recovery, the shear moduli measured will vary, depending on the way in which the stress has been varied, and even if a long time for recovery is given, the recoveries are never complete (as already explained) on account of the starch. The variation of modulus with stress history thus produced is known as "elastic hysteresis." If the conditions of experimentation are carefully standardised a reproducible modulus can, however, be obtained.

Structural Viscosity and Work-Hardening.—The viscosity of a true fluid is a constant, irrespective of the magnitude of stress and strain. This, however, is not so for dough, the viscosity of which falls with increasing stress ("structural viscosity") and rises with increasing strain (the latter phenomenon is akin to "work-hardening" in metals). In quoting the viscosity of a dough it is therefore necessary to specify the conditions of stress and strain under which the measurement was made.

It is clear that in order to measure the shear modulus and viscosity of a dough by extension and subsequent recovery, three possible methods are available:

(1) The shearing stress and time of extension may be fixed, and the deformation measured.

(2) The shearing stress and deformation may be fixed, the time of extension being measured.

(3) The deformation and time of extension may be fixed, and the shearing stress measured.

All three methods have been used, each being suitable for certain purposes, but for the greater part of the experiments whose purpose was to relate the physical properties of doughs with their baking qualities, the measurements were made by means of method (1).

In using this method, as just described, it is preferable to work at comparatively small strains, since even if a compensating device is in use to allow for the thinning of the cylinder, dough cylinders do not extend very uniformly at high strains, and it is not possible to keep a control on the shearing stress. A convenient way of investigating the region of high strains is to employ method (3), extending the sample for a fixed time at constant rate and letting the stress build up as it will. In this way, enormous extensions (500% in a normal dough) can be obtained without tearing the sample. Under these conditions a protein structure is built up in the dough quite unlike anything existing at low stresses and strains. This region has not yet been fully investigated, but although there does not appear at the moment to be any evidence that data obtained in it will differentiate flours any more exactly than those got from the present range, it seems very probable that a further understanding of the process of work-hardening will result, and this should throw some light on those aspects of the shortness problem which are still obscure.

Experimental

PREPARATION OF THE TEST PIECE.—In the early stages of this work serious difficulties were encountered when attempting to obtain reproducible measurements on different test pieces from the same dough and much time was spent in developing a technique. The method finally adopted has proved on the whole to be satisfactory.

It is necessary to make as homogeneous a dough as possible, and machine mixing of the flour and water gives the most satisfactory results. The longer the time of mixing the more homogeneous is the finished dough, but excessive mixing has a marked effect on the dough's physical properties.

From the data in Table I [5] it can be seen that excessive mixing considerably lowers the viscosity and modulus of the dough but that these increase again on resting. Such treatment, however, permanently lowers the tensile strength of the dough.

[5] Some of the data quoted in this paper are reproduced from the J. Phys. Chem. (see Halton and Scott Blair 1936 and 1936a).

It will be shown later that the general tendency on ageing a dough is for both viscosity and modulus to fall. It is only after prolonged mixing that the opposite effect more than compensates for this fall, producing a rise in both properties. Thus it appears in Table I that after 3 minutes' mixing the effect of standing is the opposite to that produced by standing after 12 minutes' mixing.

TABLE I

THE EFFECT OF MIXING ON THE VISCOSITY AND MODULUS OF THE DOUGH

Time of mixing	Time of ageing after mixing	Viscosity	Modulus
minutes	*minutes*		
3	0	6.38×10^6	4.29×10^4
	30	4.92×10^6	3.94×10^4
	60	4.04×10^6	3.73×10^4
12	0	2.92×10^6	3.02×10^4
	30	3.55×10^6	3.41×10^4
	60	3.51×10^6	3.50×10^4

The sample of dough is transferred from the mixer to a "gun" consisting of a hollow metal cylinder 5 cm. long by 2.5 cm. in diameter, fitted with a plunger. To the bottom of the cylinder is fitted a solid piece of metal drilled with a hole 3.5 cm. long and 0.5 cm. in diameter. This gun, into which the dough is placed by means of a spatula, thus avoiding handling it, is too big to fit conveniently on the apparatus and therefore the extruded dough cylinder is squeezed directly into a second smaller gun 15 cm. long and 1 cm. diameter which is fitted with an end-piece similar to that of the first gun. The dough is forced from this gun straight on to the surface of a bath of mercury.

The plungers of these guns were in the first case worked by hand, but it was found that the pressure that had been applied by hand was excessive and irregular and affected the dough's physical properties in an erratic manner. A system of pulleys and weights was therefore used, and provided the weights were small, the properties of the prepared cylinder were approximately independent of the weight used. The importance of a carefully standardised use of these guns cannot be too strongly emphasized. During extrusion the dough cylinder swells, this swelling being in general greater for "strong" than for "weak" flours. The swelling is, however, affected by a number of factors and its exact connection with dough quality is not yet perfectly understood.

THE EXTENSIMETER.—This instrument which is a development of those described in earlier papers (see Schofield and Scott Blair 1932, and 1933a) is shown diagrammatically in Figure 1.

A dough cylinder A, about 10 cm. long by 0.7 cm. in diameter, made as described above, is floated in a mercury bath. The ends of this cylinder are fastened to cork "chairs" connected by very fine cotton threads to two small scales B, which are observed through low-power microscopes C. The smallest divisions on the scales are 0.013 cm. in length and readings to one-tenth of this can be estimated with a fair degree of accuracy. To one scale is fastened a steel spring D, the other end of which is securely attached to the framework of the apparatus; the other scale is connected by cotton to a small winch E, which can be wound either by hand or by a small motor.

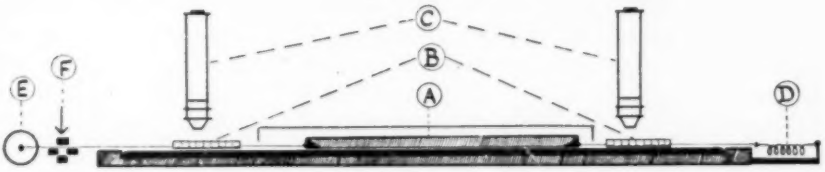

Fig. 1. Diagram of extensimeter.

During experiments the dough is protected by a cover, the felt lining of which is damped to provide a humid atmosphere, preventing drying of the dough surface.

When the winch E is wound up the dough and scales move to the left and this extends the spring D. The dough is therefore subjected to a stress, the value of which is proportional to the extension of the spring and inversely proportional to the cross-section of the dough cylinder. The spring was calibrated by noting the extension caused by hanging weights of various sizes from it. The diameter and initial length of the dough cylinder are measured with calipers.

If the spring is stretched by x scale units and it takes k grams to stretch it one scale-unit the tensile stress on the dough per unit cross-section is $\frac{gxk}{\pi r^2}$ dynes where r = radius of the dough cylinder and g is the acceleration of gravity = 981 c.g.s. units.

$$\therefore \text{ Shearing stress } S = \frac{981xk}{3\pi r^2}.$$

From this equation it is possible to calculate the necessary deflection of the spring for any required stress.

When the dough is in position the winch is adjusted so that the cotton threads, scales, and dough cylinder are just taut and in a straight line. Readings L_1 and R_1 of the scales are taken. The winch is then rapidly wound up until the spring is deflected by the required amount, as noted by the reading R_2 of the scale attached to it, to produce the

desired stress S. By adjustment of the winch to take up the stretch of the dough cylinder the scale R is kept at the reading R_2 for a given length of time (t) say one minute, at the end of which time a reading L_2 of scale L is taken and the stress immediately released. After the dough has relaxed as far as it will (after about 3 mins.) the winch is adjusted so that the cotton threads *etc.* are just taut and the scale R is at its original position R_1. A reading L_3 of scale L is then taken.

The viscous stretch of the dough is then equal to $\frac{k_1(L_3 - L_1)}{l_1}$

where k_1 is the conversion factor for scale readings into centimetres and l_1 is the original length of the dough cylinder

$$\eta = St \Big/ \frac{k_1(L_3 - L_1)}{l_1}.$$

It should again be emphasised that the viscosity is changing during the process due to work-hardening, so that η is the *mean* viscosity over the period of the extension.

The elastic recovery of the dough cylinder on removal of the stress is

$$\frac{R_1[(L_2 - L_3) - (R_2 - R_1)]}{l_3}$$

where l_3 = the final length of the dough cylinder.

$$\therefore n = S \Big/ \frac{R_1[(l_2 - l_3) - (R_2 - R_1)]}{l_3}.$$

It is thus possible to obtain measurements for the viscosity and modulus of the dough from a single experiment.

When a dough cylinder increases in length its cross-section decreases and since with the above apparatus the deflection of the spring is kept constant the stress per unit area on the dough increases with increasing strain. This error is not serious if the strains are kept small.[6]

Relation between the Physical Properties and Baking Value of Dough

WATER ABSORPTION.—When comparing the quality of a number of flours in the bakehouse the water content of each dough is adjusted to suit the particular sample being tested. This procedure is also necessary when comparing the physical properties of different doughs and the problem of fixing water absorption in terms of some physical property had therefore to be investigated.

In assessing water absorption the baker relies on his sense of touch and he appears to be principally guided by the extent to which the

[6] One of the authors (P. Halton) has developed an apparatus for working at constant stress, irrespective of strain. This will be described in a later paper.

dough sticks to his hands. On the assumption that as much water as possible is added to the dough without causing undue stickiness was based our first attempt to fix absorption by measuring some physical property.

Of suitable methods for measuring the stickiness of doughs that of Bouyoucos (1932), originally devised for use with soils, was found the most satisfactory. It was not, however, found possible to make the measurement sensitive enough as a means of fixing water absorption and the question of the use of some other physical property less obviously connected with correct water absorption had to be considered.

For the examination of the change in the viscosity and modulus of dough with changing absorption a number of flours were used. The data obtained on three of these, a Manitoba, a Plate and an Australian, are given in Table II. Each flour was examined at bakehouse absorption and at four other water contents, $\pm \frac{1}{2}$ gallon and $\pm$ 1 gallon per 280-pound sack of flour. Each dough was fermented for 4 hours, at the end of which time samples were taken for viscosity and modulus measurements. In Figure 2 curves are drawn showing the relationship between these two properties for each of the flours at the five water contents.

TABLE II

EFFECT OF WATER CONTENT ON VISCOSITY AND MODULUS OF DOUGHS

Flour	Water content				
	−1 gal.	−½ gal.	Normal	+½ gal.	+1 gal.
No. 1 Manitoba					
Viscosity ($\times 10^6$)	10.0	7.5	5.8	4.8	4.1
Modulus ($\times 10^4$)	4.1	3.6	3.1	2.6	2.1
Barusso Plate					
Viscosity ($\times 10^6$)	15.0	8.3	5.7	4.4	3.5
Modulus ($\times 10^4$)	4.6	4.0	3.4	2.8	2.2
Australian					
Viscosity ($\times 10^6$)	7.5	4.8	3.5	2.8	2.3
Modulus ($\times 10^4$)	5.5	4.6	3.6	2.6	1.6

The three doughs of particular interest are the "normal" doughs made up with the normal bakehouse absorption. These three doughs differed considerably in viscosity (3.5 to 5.8) but to a very much smaller extent in modulus (3.1 to 3.6). This suggests that absorption is closely connected with a constant modulus, a standard which has been found very convenient for use when comparing the physical properties of doughs made from a variety of flours.

SPRING.—This experiment also showed that by adjustment of water content it was possible to make doughs from each of the flours

to have either the same viscosity or the same modulus but that by no such adjustment could the viscosity-modulus relationship be made the same for the three flours without making very wide differences in viscosity itself. If the viscosities were adjusted to the same value then the Manitoba dough had the lowest and the Australian dough the

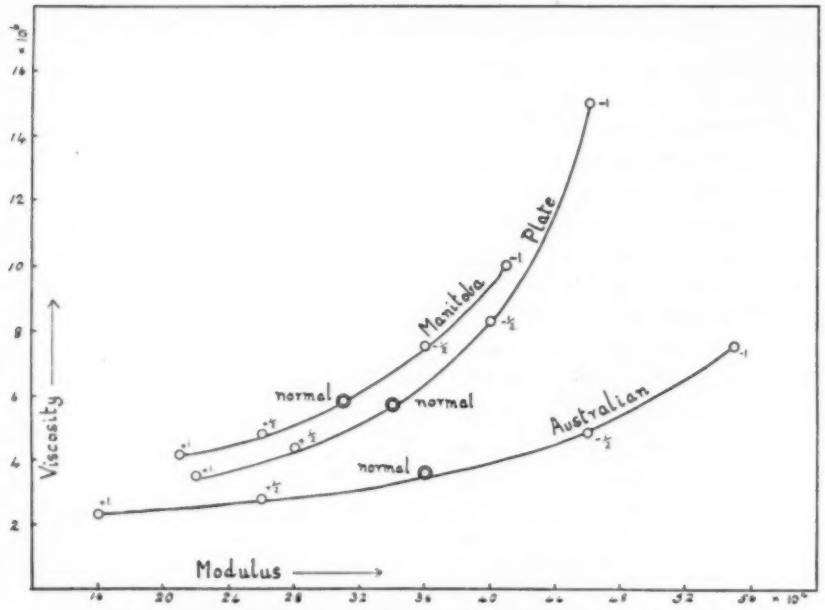

Fig. 2. Illustrating relationship between viscosity and modulus of dough.

highest modulus and if the moduli were the same then the Manitoba dough had the highest and the Australian dough had the lowest viscosity. This suggested that the ratio η/n, which has been called by Maxwell the "relaxation time" (see Schofield and Scott Blair, 1933a), was of fundamental importance in determining the baking quality of flour. The numerical value of η/n is not independent of water content and it has been found most convenient to compare relaxation times at identical values of n.

Of all the impressions gained by a baker during the handling of dough the one to which he pays most attention in forming his judgment of quality is probably "spring," *i.e.*, the recovery of the dough after deformation.

The "spring" of a dough is considered by the authors to be closely associated with relaxation time. The connection can be demonstrated as follows. Imagine two helical springs with straightened ends held loosely together by a clamp (Figure 3). If the system is extended, held for a moment under stress, and then allowed to recover, the pro-

portion of the initial extension which cannot be recovered will depend on the amount of "slip" at the clamp. The amount of slip depends on the balance between two factors, the friction at the clamp which resists slip, and the force set up in the extended springs (which depends on the toughness of the springs) which favours slip. For little slip to take place, and therefore good recovery of the system after extension, the friction at the clamp should be high relative to the toughness of the springs.

Fig. 3. Illustrating the spring of dough.

In the case of flour doughs the friction corresponds to viscosity and the toughness of the springs to modulus. Good spring (*i.e.*, recovery) is therefore associated with a high viscosity-modulus ratio and poor spring with a low value for this ratio. Moreover, since relaxation time is given by the ratio of viscosity to modulus, good spring is associated with a high relaxation time.

The significance of the importance attached by bakers to spring is thus apparent when its connection with relaxation time is realised. A high viscosity-modulus ratio means that when the dough is inflated during fermentation a large proportion of the extension is elastic, therefore the dough structure is more stable since less viscous flow (with its accompanying change of shape and breakdown of cell structure) results.

SHORTNESS.—While the spring factor in dough is a most important, perhaps the most important, factor in determining baking quality, a measure of relaxation time alone is not sufficient to classify flours.

Many flours are met with in the bakehouse that are called "short" on account of the ease with which they tear under bakehouse manipuplation. With all doughs there is probably a tendency to tear, although it only becomes apparent to the baker when at the stage when the dough tears easily. Bad shortness is obviously a controlling factor in the quality of bread that can be produced from any given flour, but it is probable that the tendency to tear, even in doughs which the baker does not consider short, has an important influence on the gas retaining capacity of the dough and on the crumb structure. This is suggested from the results obtained by adding increasing amounts of some shortening agent, such as fat, to a dough. The effects produced are progressive and result in decreased loaf volume, coarsening of crumb structure, and a torn crust. Small doses, while not making the dough obviously short, do have some effect on loaf volume and crumb structure.

Early attempts by the authors to measure shortness consisted in measuring the *extent* to which dough cylinders could be stretched at constant rate before snapping, but the "extensibilities" (ductilities) so measured failed to classify flours in order of their shortness as noted in the bakehouse. Direct methods of measuring "tensile strength" (breaking *stress* per unit area) could not be used, due to the impossibility of measuring the cross-section of the dough at the point of rupture. A method of assessing shortness has been developed, however, out of an observation that "short" doughs extruded from the gun faster than non-short doughs when both were of similar viscosity as measured on the extensimeter. The time of extrusion from the gun is mainly determined by the viscosity of the dough, a viscosity, however, which is markedly less than that measured on the extensimeter since the stresses applied in the gun are far greater than those on the extensimeter. This observation suggested a connection between shortness and structural viscosity (*i.e.*, the fall in viscosity with increasing stress).

From observations on the manner in which various materials pour out of a narrow tube, Schofield and Scott Blair (1935) concluded that in the case of materials which show both solid and fluid properties, the rate of decrease of viscosity with increasing stress may be the determining factor in their tensile strengths. This idea has been extended by the present authors (Halton and Scott Blair, 1936a) who point out that when a piece of dough is extended until it breaks, a heterogeneous structure is formed which can be seen even with the naked eye. This means that in the cross-section of the dough there are points of extreme toughness and also points of weakness about which the tougher "fibres" slip.

In a short dough this heterogeneity is exaggerated, so that as the stress rises the slip along the cleavage planes becomes rapidly more serious and the measured viscosity falls. An alternative way of expressing this is to point out that the shear angle becomes more and more distorted, a phenomenon known to have drastic effects on the brittleness of metals. As extreme cases we may consider a liquid as infinitely extensible, the shear planes being infinitely thin, the shear angle undistorted, and the viscosity independent of stress. The metal is, in a sense, the extreme case of shortness. The viscosity is infinite at low stresses, falling to a finite value at a stress which may be hardly below the tensile strength. Such flow as does occur takes place along widely separated shear-planes, and the change in measured viscosity between zero-stress and the stress at which rupture occurs is enormous.

It is appreciated that "shortness," which depends not only on

tensile strength (a stress) but also on ductility (a deformation), is probably at least as much affected by work-hardening as by structural viscosity. This aspect of the problem is being further investigated, but it is already clear that at strains which are not too large and at stresses of the order used in the experiments, high work-hardening and high structural viscosity generally go hand in hand.

This is not surprising since during the stretching the fibres are aligned, resulting in work-hardening, and at the same time any increase in stress will result in the breaking of the junctions between them and therefore in decreased viscosity. When such a junction breaks a rent is formed and the previously extended fibres contract, making the rent worse. At such points, therefore, an elastic extension changes to a viscous slipping of the fibres and at the same time the release of stress helps to cause further increased extensions. The net result is to cause a fall in the measured viscosity of the whole sample.

The method of measuring structural viscosity consists in measuring the viscosity of the dough at two different stresses. The time of extrusion of a standard quantity of dough from the gun can be taken as a measure of viscosity at high stresses, but it has been found preferable to obtain two measurements on the extensimeter. Viscosities have been so measured at stresses ranging from 500 to 4,000 dynes/cm.^{2}, but most of the data to be given later in this paper were obtained at stresses of 1,000 and 2,000 dynes/cm.^{2} A measure of structural viscosity is then given by the ratio η_{1000}/η_{2000}.

Effect of Temperature.—Experiments have shown that the viscosity of a typical dough falls by about 10% per degree centigrade rise in temperature and the modulus by about 5%. In view of the large effect of temperature, a constant temperature room was built, and much of the later experimental work recorded in this paper was done in it.[7]

Since viscosity falls about twice as fast as modulus with rising temperature the all-important relaxation time (viscosity-modulus ratio) is higher the lower the temperature. This important fact supports the view held by some bakers that dough should be fermented at as low a temperature as is consistent with the satisfactory working of the yeast.

Ageing and Fermentation of Dough.—Dough is fermented with yeast primarily for the production of carbon-dioxide to inflate it. If, however, a series of replicate yeasted doughs are fermented for varying times before going to the oven it is found that the quality of bread produced varies with the fermentation time. Too short or too long a fermentation time produces poorer bread than when the time is

[7] Adequate ventilation is essential to eliminate the danger of poison by mercury vapor: see Stock (1926).

correct for the particular flour being used. It is thus pertinent to inquire as to the connection between optimum fermentation time and the changes that take place in the dough's physical properties.

Experiments have shown that both the viscosity and modulus of doughs fall during fermentation, and that since the viscosity falls more rapidly than the modulus, the relaxation time (η/n) also falls.

The fall in relaxation time would suggest that bread quality should deteriorate as fermentation progressed, and the reason for the improvement in quality during the ripening or early stages of fermentation cannot be explained in terms of those physical properties of dough that have so far been examined. Although undoubtedly there are other physical properties which may be important, it is possible to explain the facts of fermentation or dough ripening on purely mechanical lines.

Before a good loaf can be made the necessary cell structure has to be built up in the dough and this cell structure must be determined by the number and distribution of the yeast cells. Now, normal bakehouse mixing is comparatively crude, and, in consequence, this distribution is probably anything but uniform. Owing to the activity of the yeast cells the dough swells, and this probably helps to spread the yeast. This is further helped by the baker's knocking back and moulding the dough, while the multiplication of the yeast cells during fermentation again increases the number of gas-producing centres.

If the above picture is correct, and it is the building-up of the necessary cell structure which determines how much fermentation is required for any flour to give its best bread, then it should be possible to cut down this time by using more yeast and/or more thorough mixing. That this is so is well known (see Fisher and Halton, 1936).

During fermentation two processes, that of building-up of structure by the gas production and that of fall-off in physical properties, go on side by side. With strong flours the high initial η/n ratio means that good quality bread can be produced over a large range of time, whereas with weak flours the ratio is low to start with and the subsequent fall in value means a rapid deterioration. Thus, the tolerance of a flour to increasing fermentation time is dependent on the physical properties of the dough and the change in those properties during fermentation.

It has usually been considered that the change in dough quality during fermentation, known as dough-ripening, was due to chemical actions associated with yeast activity. This, however, does not appear to be so, since experiments have shown that the presence of yeast has but little effect on the dough's physical properties, or on the way these change during fermentation, and suggest that the rôle played by yeast in a fermenting dough is largely that of a gas-producing agent.

The recent work of Erbring (1936) on "fibrosity" ("Spinnbarkeit") suggests that there are flow properties which are of great importance in modifying the behaviour of materials but which are not directly related to viscosity and probably not to elastic moduli. The possibility of the importance of such properties on dough quality and the possible influence of yeast on them cannot therefore be disregarded.

CHOICE OF EXPERIMENTAL CONDITIONS AND CORRELATION WITH BAKING.—The technique employed in measuring the viscosity and modulus of doughs has already been described, but certain important details connected with these measurements must now be discussed.

As has already been mentioned, the viscosity of dough is not a constant but depends on the stress and strain to which the dough is subjected. For exact correlation with bakehouse behaviour these stresses and strains should approximate to those operating in the fermenting mass. Unfortunately these latter cannot be directly assessed and the correct experimental procedure can at the moment only be arrived at by trial and error.

Ranges in stress from 500 to 4,000 dynes/cm.^{2} have been experimented with [8] and times of application of these stresses have varied up to 15 minutes. However, when dealing with flours of considerably varying character, conditions of experiment have to be fixed that are applicable to all samples, and it has been found that stresses of 1,000 and 2,000 dynes/cm.^{2} applied for one minute are the most satisfactory to use with the apparatus at present available. When measuring the modulus, the dough is allowed to relax for 3 minutes, as in this time most of the elastic after-effect is completed.

The flour to be examined is made into dough with sufficient salt solution to give the required consistency. The dough is kept in a stoppered bottle and samples taken at intervals for examination. At all stages very great care is taken to prevent drying, as a surface skin on the dough has a very marked influence on the measurements. Curves are drawn relating both the viscosity and the modulus to the age of the dough, and from these curves the viscosity for any particular modulus can be read off. It has been found convenient to use a modulus of 1.0×10^4 c.g.s. units. A selection of data so obtained is given in Table III. These data were all obtained in a constant temperature room kept at 80° F., and the figures are not directly comparable with those given earlier in this paper which were obtained at different stresses and much lower temperatures. All the flours in this table were laboratory milled with the exception of the imported Canadian sample.

The two No. 1 Manitoba flours were unsatisfactory samples, and in

[8] Schofield and Scott Blair (1937) have worked up to stresses of 15,000 dynes/cm.^{2}, using a different technique.

TABLE III

RELATION BETWEEN VISCOSITY AND MODULUS FOR DIFFERENT DOUGHS

Laboratory No.	Flour type	Relaxation time, Spring (η/n)	Structural viscosity (Shortness) $\left(\frac{\eta_{1000}}{\eta_{2000}}\right)$
S424	Imported Canadian	102	1.3
NC 946	No. 1 Manitoba	85	1.3
NC 1011	No. 1 Manitoba	84	1.3
NC 958	No. 2 Manitoba	91	1.4
NC 1013	No. 2 Manitoba	89	1.2
NC 1005	No. 3 Manitoba	58	1.3
NC 1010	No. 4 Manitoba	52	1.2
NC 995	South Australian	55	1.5
NC 997	South Australian	50	1.9
NC 998	Western Australian	50	1.9
NC 1012	South Australian	38	1.6
W 152/8	English	79	1.4
W 151/7	English	56	1.6
W 167/8	English	28	2.0

the bakehouse were very slightly inferior to the samples of No. 2 Manitoba. The No. 3 Manitoba was also a poor sample of its type and its much lower spring figure of 58 compared with about 90 for the No. 2 Manitoba samples is in keeping with its much poorer quality in the bakehouse. The No. 4 Manitoba was one of a batch of Nos. 4, 5, and 6 Manitobas which were all badly frosted. The flours were all poor in the bakehouse, the doughs being gummy and short. With none of these flours did the structural viscosity figure agree with the excessive shortness.

Of the four Australian flours No. 997 and No. 998 were both very short while the other two were only slightly short and yielded much better bread.

The three English samples were interesting in that they were special experimental wheats. W152/8 was an extraordinarily good sample and quite unlike ordinary English. Its high spring figure and low shortness figure are quite in keeping with its quality. W151/7 was quite good for all-English wheat flour and showed only slight shortness. W167/8, however, was a typical poor English sample and was very short.

These samples are sufficient to show the close connection between baking quality and the two physical properties of relaxation time and structural viscosity. At this stage exact correlation is hardly to be expected owing to the complexity of the system being examined and the difficulty of measuring shortness. As has already been stated, the value of η/n depends on the stress at which it is measured and the

correct value of stress to use can only be determined by experience. The problem of shortness is even more complicated since shortness appears to depend partly on the structure of the dough and it is much more marked in an inflated dough than in the same dough when all the gas has been knocked out of it. For this reason while the structural viscosity figure agrees with the estimated feel of shortness in the non-inflated dough, it does not always agree with the shortness shown up in the baked bread. There is also the "fibrosity" factor to be considered, but measurements of this property, which may well be important in shortness, have not yet been made for doughs.

Conclusions

Subject to adequate capacity to produce gas, the baking quality of a flour depends on the physical properties of its doughs. This has been known for some time, but earlier workers have attempted to assess baking value by comparison of bakehouse results with the results of tests which measure some unknown and complex mixture of physical properties. The work necessary to mix a dough, or to blow a bubble in it, is a very complex function of viscosity and elasticity modulus, and may involve other properties as well. Baking value also depends on a complex mixture of properties, and it is difficult enough to sort these out into their simple components without attempting correlation with other equally complex systems. It is therefore advisable in the first instance to attempt to assess baking quality in terms of such relatively simple properties as viscosity and shear modulus. Although it is realised that properties other than those which have been measured may be of importance, experiments have shown that the two most important factors in bread-making are spring and shortness, and that the former is adequately measured by the ratio of viscosity to a power of the shear modulus, while the latter is generally in fair correlation with the rate of fall of viscosity with increasing stress. There are, however, exceptional cases where shortness in the dough does not appear to be associated with high structural viscosity, and some further factor is doubtless involved. The results of very many tests have been sufficiently encouraging to convince us that by measuring these fundamental properties a complete understanding of the physical aspects of dough fermentation will finally be reached. Since viscosity and modulus vary, depending on the stress and strain conditions of the experiment, these conditions must be fixed so as to tally as closely as possible with those prevalent in the baking process.

To summarize, we may classify the properties of a dough requisite for good bread-making as follows:

When made with the right amount of water to give a suitable shear modulus the viscosity should be as high as possible.

For adequate extensibility, the viscosity should not fall too rapidly with increasing stress.

The rate of fall in viscosity with addition of water and ageing during fermentation should be slight, in order that there may be adequate tolerance to variations in absorption and fermentation-time in the bakehouse.

The importance of such factors as extent of elastic hysteresis, rate of elastic after-effect, "fibrosity," *etc.*, are not yet fully understood. Further work is required to elucidate them.

It is clear that as technique improves it becomes increasingly possible to determine the above properties with accuracy. When this process is developed, it should be possible to obtain a valid estimate of the commercial value of a wheat or flour without recourse to the baking test, which is so much influenced by personal factors.

Summary

The baking quality of a flour has been found to depend on the physical properties of the dough and a picture is given of the mechanism of dough behaviour during fermentation based on these physical properties.

The two properties which are of chief importance are viscosity and elasticity modulus and methods of measuring these in absolute units have been devised.

The viscosity and elasticity modulus of dough are not constants but depend on the magnitude of the stress and strain to which the dough is subjected. They also vary with the water content, age, and temperature of the dough.

The baking quality of a flour depends primarily on the spring and shortness of the doughs. The spring of dough depends on the relationship between viscosity and elasticity modulus, the higher the η/n ratio the better the spring. Shortness in doughs is connected with structural viscosity, *i.e.*, the rate at which viscosity falls under increasing stress.

Note

Since writing this paper, the authors have read an article published in this journal by Bohn and Bailey (1936) in which a modification of the Schofield and Scott Blair method of measuring plastic and elastic properties of flour was described. The partial correlation between the maximum load built up when a cylinder of dough is extended under standardised conditions and the quality of the dough to stand heavy handling in the bakehouse is most interesting, and does not in any way modify any of the conclusions reached in the present paper. It is clear from our work that baking value depends on something much

more complex than a single stress value. Flour dough does not have a constant mobility over any appreciable range of stress, so that the use of the Bingham equation is precluded (see full explanation, Schofield and Scott Blair (1932)).

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EVALUATION OF MALT FOR BREWING

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The problems connected with the evaluation of barley and malt intended for brewing have been the subject of extensive studies for a long time. Numerous discussions on this matter are published in the brewing literature every year.

With the return of beer and the resulting demand for improvement in the quality of malting barley, it is but natural that the question at present is receiving close attention in this country. Although it is not advisable to separately deal with the qualities of malt and of barley, since the former to a great extent are dependent on the latter, it is proposed to confine this discussion to the problem of evaluating the qualities of malt intended for brewing, on the premises that the barley from which the malt is produced meets the requirements specified in this country for a malting barley.

In order to gain information as to the properties of a malt, this is subjected to a standard analysis. This examination, as carried out, for example, by standard methods adopted by the American Society of Brewing Chemists, is more of commercial than of technical importance, because malt is of such a complex composition that we are unable, with the present state of our knowledge, to assess its true qualities. In spite of its shortcomings, an examination of a malt affords some indication as to its value and subsequent behavior in the course of brewing procedure.

Physical Examination of Malt

Let us consider first the physical analysis of a malt.

WEIGHT DETERMINATION: This examination includes the determination of bushel weight, 1000 kernel weight, assortment (size), acrospire growth, mealiness, percentage of foreign seeds, broken kernels, and the presence of mold. Bushel weight and 1000 kernel weight largely depend on that of the barley from which the malt is made. These measurements decrease in the process of transforming barley into malt and are therefore under certain circumstances usable as measures of modi-

fication of the barley. The weight values obtained, especially that for the 1000-kernel weight, also may indicate the type of barley (Manchurian, two-rowed, Pacific Coast barley) that was used for the production of the malt in question; in which case the experienced observer also considers the botanical characteristics. Provided a malt is sound, mellow, well modified, with normal moisture content and high enzymic activity, and one that has been produced from a malting barley that meets the standard requirements, there appears to be no justification for the objection sometimes raised against a high bushel weight. On the contrary, other conditions being equal, a high bushel and a high 1000-kernel weight indicate a high percentage of extract and consequently good quality.

ASSORTMENT OR SIZE: A determination of the uniformity in size of the malt kernels (assortment) deserves more consideration in judging malt than this important characteristic has received heretofore. The uniformity of the malt obviously is dependent on that of the corresponding barley because well sized barley not only will yield uniform malt kernels but also will steep, grow, and modify uniformly.

From a sizing standpoint, it is most desirable that the major portion (80% or more) of the malt sample under examination be retained on two adjacent sieves, rather than being uniformly present on several sizes of sieves, because the brewer on noting the mesh of the sieves on which the bulk of the malt is retained, is able to set his mill for proper grinding. For the brewing process all of the kernels should be well crushed and none should pass unbroken. To do so reduces the yield of extract. On the other hand, too fine grinding causes compact settling of the grains in the mash tun which in turn interferes with proper draining of the wort.

KERNEL MODIFICATION: A very important quality of the malt is its modification. This manifests itself by a marked change in the physical condition of the endosperm. The hard texture of the barley kernel is transformed into the relatively friable texture of the malt. The practical maltster gauges modification by observing the growth of the acrospire and the increase in the friability (mealiness) of the malt. To determine the extent of barley modification, the technical analyst applies the growth and mealiness tests. A well-modified sample of malt will have an acrospire growth to the extent of three-quarters of the kernel length in about 80% of the grains. There should be very few overgrown kernels since this is evidence of over-modification. A transverse cut of the kernels of malt should reveal that over 90% are mealy (soft and chalky) and only a small percentage are glassy kernels (hard and vitreous).

Chemical Analysis of Malt

A chemical analysis of malt calls for, among other things, a determination of moisture and of the yield of extract. A high moisture content (over 6%) is not desirable not only from an economical standpoint but also because this condition may cause difficulties in brewing such as in grinding, draining of the wort, and subsequent clarification of the fermented beer. Yield of extract obviously is an important item in malt, but it must be borne in mind that the brewer to some extent is willing to sacrifice a high extract in favor of other qualities such as good modification and high enzymic power, which factors are apparently closely identified with the protein stability of the resultant beer. The laboratory extract generally is a few per cent higher than the brewery yield, but, depending on the equipment and mashing procedure employed, the brewery yield under the most favorable conditions, may reach or even exceed the laboratory yield. Laboratory extract, however, gives the brewer valuable information from which he may be able to estimate the yield to be expected under the conditions prevailing in his plant.

In addition to extract, a number of other tests, mainly qualitative, are carried out on the laboratory wort. The odor of the mash and color of the wort should correspond to the type of malt offered. The speed of filtration and degree of clarity give some indication as to modification of the malt and its subsequent behavior in the course of brewing operations. The conversion test informs the brewer as to the relative amount of diastase (and apparently also other enzymes such as protease) present, and as to the speed of conversion to be expected in actual mashing. If necessary a quantitative estimation of the diastatic power can be made.

As can be seen from this brief discussion, a technical malt analysis reveals a number of important facts which are of practical value to the brewer. It is recognized, however, that the information given by these tests is inadequate, and, at times, even with a favorable analysis report, trouble still may be encountered in filtration, with the break in the kettle, in fermentation and clarification. Furthermore, the present commercial analysis still leaves the brewer completely in the dark with regard to the character of the malt proteins and their influence on foam stability, palatefulness, gushing, *etc.*

Progress in our knowledge regarding the chemical composition of malt, however, has been made in recent years. Suggestions have been made by a number of investigators with a view towards improving our criteria in judging malt quality, particularly with regard to the question of modification. For example, the physical methods of estimating modification are open to serious objections. The method of malting exerts a

considerable influence on the growth of acrospire. When germination proceeds at high temperatures growth may outstrip enzymic changes, whereas the elimination of air, as, for instance, in the CO_2 process of malting, restricts growth without exercising a corresponding effect on the enzymic activity.

Mealiness of kernel as judged by the cutting test, likewise, has its shortcomings; a malt which has been modified completely in the course of germination, may subsequently become vitreous during kilning if the temperature is raised too rapidly when the moisture content of the green malt still is relatively too high.

A method widely practiced in Germany for estimating modification is the sinker test; this depends upon suspending 100 kernels in water and after the lapse of a definite number of minutes counting the number of floaters and sinkers. The method repeatedly has been criticized adversely, and Eckhardt (1935) published a review of the subject. It appears that the inner air present in malt, the greatest portion of which is contained in the air sack formed by the growth of the acrospire under the husk, makes the kernel float upon water. This explains the general relationship between the number of floating kernels, length of acrospire, and modification. However, any injury inflicted to the air sack, which may be caused by close polishing of the grain, will make the kernels sink. This obviously renders the procedure unreliable.

With respect to hazy running laboratory worts, it has been observed repeatedly that, in many cases, this has nothing to do with modification and may be due to other causes, such as varietal and seasonal influences.

For the purpose of obtaining a more comprehensive insight into the nature of modification, a number of chemical methods were suggested, so that the " chemical modification " of malt might be determined in addition to its " physical modification."

The term " modification," as used today, comprises all the physicochemical changes which occur when barley is transformed into malt. It refers particularly to the development of enzymes and to the progressive degradation of the colloidal carbohydrates and proteins, and is brought about by the enzymes formed. The composition and properties of malt, and of the resulting wort and beer, in a great measure are dependent upon the extent to which these substances are broken down into simpler compounds. The enzymic changes manifest themselves in an increase in soluble extract, soluble nitrogen, and other soluble substances.

Proposals have been made to use these criteria as measures of modification, but some of these data are considered to be of little significance. Thus, determination of titratable acidity, of buffer capacity, and of cold water soluble extract, seem to show little variation between individual malts. The last mentioned test is commonly employed in England and

affords a rough measure of the total enzymic activity. Some English brewers, however, object to high cold water extract as this seems to indicate "forcing" (accelerated germination at high temperature). The well-known method based on the difference in yield obtained from fine and coarse grinding also is open to criticism; in the past its significance has been overrated. Various factors affect the results: experimental error in determining extract, amount of diastase present, moisture content of malt, *etc.* To improve this method Kolbach (1935) suggested changing the specifications for determination of extract in coarse grinding, in that the sample should be ground to 25%, instead of 40%, fineness. Piratzky and Rehberg (1935) based their method of evaluation of malts on a similar principle, *i.e.*, on the difference in yield obtained between a highly intensified and a less intensive method of mashing. Hind and Hamnett (1935) proposed using the "extract index" as a measure of modification. This method depends upon comparing the potential extract predicted from the barley by means of Bishop's formula (set forth in the important work on barley by Russel and Bishop, 1933), with the actual extract obtained from the corresponding malt, when the barley was malted under standard malting conditions.

Quite recently Hartong (1936) employed the following procedure, which also is based on the extract principle, for estimating the character and modification of malt. Mashes were made for one hour at 25°, 45°, 65° and 85° C., and the extract yields were calculated and expressed in percentages of yield obtained from the same malt by the regular Congress method. The solubility of the malt at different temperatures gave some indication as to its character. The average of the results obtained at these four different temperatures was found to range from 60.9 to 69.5% for the eight malts examined. The author proposes to deduct 60 from the values thus obtained and to call the figure in excess of 60 "modification number." On this basis the following classification of malts was suggested:

Modification number	Interpretation as to modification
0 – 3½	Decreasing under-modification;
4 – 4½	Normal modification for keg beer;
5	Ideal modification for all purposes;
5½– 6	Desirable over-modification for bottle beer;
6½–10	Increasing over-modification.

It is of interest to note that two American malts which had been evaluated by this method, were considered as typically over-modified (modification number 9½) and completely unsuitable for beer production on the European continent.

At present our knowledge of the degradation of carbohydrates which

constitute the structure of cell walls of the barley, still is very meager and incomplete. The group of enzymes acting upon these carbohydrates is known under the name " cytase," and the whole process, which manifests itself in the " physical modification " when the hard barley kernel is transformed into the friable malt, is designated by the term " cytolysis." Suggestions have been made to measure by chemical means the extent of modification produced by the action of cytase or by cytolysis, for these changes most accurately measure the practical maltster's understanding of modification. Among the polysaccharides which constitute the structural tissues of the cell membrane the pentosans play an important part. It has been observed that degradation of the pentosans occurs in the course of malting and that the soluble pentosans increase considerably from barley to malt. This observation led Fink (1934, 1935) to study the analytical procedures available for determining pentosans, and to attempt the development of a new method for measuring the degree of modification, based upon the amount of soluble pentosans present in relation to the total pentosan content of malt. It seems that the soluble pentosans expressed in percentage of total pentosans increase from about 3% in barley to about 11% in malt. Investigations on the subject are being continued.

Of the chemical methods which have been suggested for determining the degree of modification of malt, the one based on the extent of proteolysis occurring in the course of malting, as revealed by the action of proteolytic enzymes, has received most consideration. This is obvious since important processes in beer production and important qualities of the final product are intimately connected with the proper degradation of nitrogenous substances. As early as in 1910, Schjerning postulated certain requirements for a normal and completed protein conversion in malt. Since then many investigations have been carried out on the proteins of barley and their breakdown in the course of malting and brewing. However, the methods employed by some of the investigators for determination of the different nitrogen fractions are too complicated for use in control work (see Lüers, 1936). For practical purposes it is sufficient to estimate the soluble nitrogen or, what is to be preferred, the permanently soluble non-coagulable nitrogen of the cold water extract, and its relationship to the total nitrogen content of the malt. This gives an indication of the degree of modification of the malt, for it has been observed that the protein breakdown approximately runs parallel with progressive modification. According to general European experience, greater protein content causes barleys to modify less readily and consequently yield less soluble nitrogen in relation to total nitrogen, than is the case with barleys of low nitrogen content.

In order to obtain further information as to the extent of protein degradation in malt, in addition to the determination of soluble nitrogen, a formol titration may be carried out on the cold water extract. This test, as is known, estimates not only the amino-acids but also all free amino groups of the polypeptides, and thus gives a measure, when compared with the solubility of permanently soluble nitrogen, of the relationship existing between the low and high molecular weight nitrogen present in malt. Both low and high molecular weight nitrogen are important constituents of wort and beer, and a proper relationship between them is necessary. The former serve as yeast nutrients, whereas the latter substances are responsible for protein haze and also for palate-fullness and foam stability. Increased protein degradation may promote brilliancy, but at the same time impair other important qualities of beer.

As a result of his extensive studies on the proteins in barley and malt, Bishop (1929, 1930, 1931) suggested using the permanently soluble nitrogen of the laboratory wort produced by the British standard methods of analysis, in relation to the total nitrogen content of the corresponding barley, as a measure of modification; the permanently soluble nitrogen being found through boiling the wort for 15 minutes. Under normal malting conditions prevailing in England, approximately 35% of the barley protein is found permanently soluble in wort for English two-rowed, and 29% for Californian six-rowed barleys. Values which are considerably higher or lower than those indicated suggest over and under-modification, respectively.

Since it is only rarely possible to compare the permanently soluble nitrogen of the wort with the total nitrogen content of the corresponding barley, and as the nitrogen content shows only an insignificant change from barley to malt, Hind (1933, 1934) and Hind and Hamnett (1935) suggested that it would be more convenient and practical to use the total nitrogen content of the malt instead of that of the barley; he then proposed to use the expression representing the permanently soluble nitrogen of the wort in percentage of the total nitrogen content of the dry malt as an "index of modification." The proper value would amount to approximately 36 for English two-rowed and 30 for California six-rowed malts, when produced under standard (English) malting conditions.

It is recognized that the determination of the permanently soluble and formol nitrogen in the cold water extract of the malt affords the most reliable means of judging the degree of modification and the extent of protein degradation in malt. However, this procedure requires an additional analysis which is rather troublesome, as it is necessary to prepare the extract at rather low temperatures to prevent further proteolysis. Following in part the suggestions of Bishop and Hind, Kolbach

(1933) considered it convenient and economical to determine the soluble nitrogen in the laboratory wort obtained in the regular malt analysis; this nitrogen value, expressed in percentage of the total nitrogen content of the malt, indicates the "degree of modification." Again for convenience, the soluble and not the permanently soluble non-coagulable nitrogen is here considered, since the determination of the coagulable nitrogen is a tedious procedure (requiring refluxing for five hours by heating in a salt bath, or boiling under pressure), and it has been found that as between individual malts, the laboratory worts show only little variation in coagulable nitrogen.

It is realized that the soluble nitrogen present in wort results from degradation of proteins both during malting and mashing. Protein breakdown in the course of mashing proceeds along lines similar to that occurring during malting, and depends both upon the amount of nitrogenous matter available and on the activity of proteolytic enzymes. The latter function, however, varies because of the restricting influence of the kilning process. Therefore, due to difference in proteolytic activity, two malts which show the same degree of modification in the cold water extract, may differ in this respect when the determination is made in the laboratory wort. However, this fact does not diminish the value of the test, since the brewer is most interested in the actual behavior of the malt in the course of brewing operations. According to Kolbach, this is revealed by the relationship of the amount of soluble nitrogen in the laboratory wort to the total nitrogen of the malt. Kolbach (1934) even went still further and suggested that proteolysis obtained in the brewery wort should be compared with the degree of modification of the malt as found through analysis of the laboratory wort. It is obvious that additional valuable information as to the extent of protein degradation in wort will be secured if estimation of formol nitrogen is included.

A large number of experiments were carried out with malts intended for production of pale lager beers in Continental Europe. Their index of modification was found to vary from 32% to 48%, with an average value of 38%. Five per cent of this value represents coagulable nitrogen expressed in percentage of total malt nitrogen. On this basis, Kolbach proposed the following tentative classification of malts:

Degree of modification	Indication as to modification
Over 41%	Very well modified
Between 35% and 41%	Well modified
Below 35%	Moderately modified

It is noted that the expressions "over-modified" and "under-modified" are omitted. According to Kolbach, such classifications would

imply a judgment of the malt, which, because of our present limited experience with this new malt criterion, must now be avoided. Later Kolbach (1935a) dealt with the influence of malting conditions on the "degree of modification."

Investigations of Schjerning (1910), Lüers (1928), Krauss (1932), and Jalowetz (1931) seem to indicate that, in general, low malting temperatures are more favorable to the formation of proteolytic and other enzymes as well as of soluble nitrogen, than high malting temperatures. It has been observed, however, that the increase in soluble nitrogen does not proceed parallel with modification at all stages of germination. Frequently during the last few days no increase in soluble nitrogen occurs, although modification may continue to proceed, as indicated by the growth of acrospire and other tests. An explanation of this phenomenon was offered by Bishop (1929) who suggested that soluble nitrogen continues to be formed during the whole process of germination, but in the later stages a considerable amount of it is reconverted to the insoluble form by the growing embryo (acrospire and rootlets). Degradation thus may be counterbalanced, and when growth is forced at higher temperatures even may be exceeded by re-synthesis. Under such circumstances a low soluble nitrogen content may result. The formation of soluble nitrogen further is influenced by the method of steeping, duration of germination and aeration, process of kilning, and—last but not least—by the variety of barley. Thus, high nitrogen and six-rowed varieties, although perfectly modified, in general produce less soluble nitrogen when expressed in percentage of total malt nitrogen, than two-rowed and low nitrogen varieties. However, despite these limitations, all the investigators agree that the method provides a useful means of judging the degree of modification and quality of malt.

TABLE I

COMPARATIVE MALT PROTEIN ANALYSES OF CONTINENTAL PALE MALT

Type of analysis	Cold water extract	Laboratory wort
	%	%
N (Malt N)	1.4 to 1.7	1.4 to 1.7
SN (Soluble N)	0.5	0.62
SN in % of N	28 to 34	35 to 41
DM (Degree of modification)		35 to 41
PSN (Permanently soluble N)	0.38	0.54
PSN in % of N	22 to 26	31 to 37
IM (Index of modification)		31 to 37
FN (Formol N)	0.15	0.2
FN in % of N	8 to 11	11 to 14
FN in % of SN	28 to 32	29 to 34
FN in % of PSN	38 to 42	34 to 40

Comparison of Continental, British, and American Malts

Data presented herewith offer a comparison between Continental (European), British, and American malts on the basis of soluble and formol nitrogen content and degree or " index of modification " as determined by the nitrogen test.

Continental analyses (average values taken from various sources) for normal pale malts are given in Table I.

The results obtained obviously will differ depending on whether the malt analysis is carried out by the British Institute of Brewing, or by the Congress method. Five English and two California malts produced in England were analyzed by Hind (1934) by both methods. The results given in Table II were obtained:

TABLE II

COMPARATIVE MALT PROTEIN ANALYSES BY BRITISH AND CONTINENTAL METHODS

Malt number		Institute of Brewing method			Congress method		
		FN in % of			FN in % of		
		N	IM	PSN	N	IM	PSN	N
English	1	1.280	39.4	26.3	10.4	51.1	27.0	13.8
"	2	1.475	36.2	24.9	9.0	44.0	23.9	10.5
"	3	1.472	35.4	24.3	8.6	41.1	24.5	10.0
"	4	1.595	33.2	24.2	8.0	38.6	24.5	9.5
"	5	1.625	29.3	24.0	7.0	37.3	24.2	9.0
California	6	1.585	28.8	21.6	6.3	34.3	23.9	8.2
"	7	1.561	25.8	21.0	5.4	34.2	21.1	7.2

The differences in the " indices of modification " obtained by the two methods are due to variation in fineness of grinding, concentration of the mash, and particularly to variation in mashing temperatures; the proteolysis is more intense in the Congress mash at 45° C. than with the English mash at 65.5° C. Judged by the English standards, malt No. 1 appears to be more fully modified than is customary, Nos. 2 and 3 are well modified, No. 4 moderately, and No. 5 under-modified. Of the California barleys, No. 6 is well modified, and No. 7 moderately modified. On the other hand, judged by the Continental standards, malt No. 1 seems to be decidedly over-modified (very low malt N), Nos. 2 and 3 very well modified, and Nos. 4 and 5 well modified. The " index of modification " of the California malts, whose low malt nitrogen compares favorably with those of English malts, show some variance by the English method, but are identical and almost close to normal by the Congress method.

The following analysis reported by Fink (1934) is of interest, since it gives a comparison between Continental and English malts as revealed by determination of nitrogen fractions in the cold water extract.

TABLE III

COMPARISON BETWEEN CONTINENTAL AND ENGLISH MALTS AS REVEALED BY AN ANALYSIS OF THE NITROGEN FRACTIONS IN THE COLD WATER EXTRACT

Malt number	Type of malt	PSN in % of malt N	FN in % of malt N
		%	%
1	Pale, Continental malt	23.9	11.3
2	Pale, Continental malt	22.6	10.6
3	Vienna, English malt for pale ale	31.0	15.6
4	Vienna, English malt for mild ale	31.2	16.1

The difference in modification between the two types of malt is striking.

Hind and Hamnett (1935) carried out some analyses on malts received from the United States. The values given in Table IV are of interest.

TABLE IV

NITROGEN MODIFICATION IN AMERICAN MALTS

Malt number	Type	1000-kernel weight, Dry basis	N in malt	PSN in % of malt N "IM"	D.P.
		Grams			*Lintner*
1	Oderbrucker	31.7	2.061	27.4	91
2	Manchuria from Minnesota	32.2	2.338	29.1	94
3	Wisconsin No. 38	24.7	2.367	28.8	170
4	Manchuria from North Dakota	32.7	2.282	24.7	93

Considering the high nitrogen content of these malts, it is remarkable that by the British method they yield a relatively high index of modification similar to that of California malts.

A number of malts analyzed in our laboratories gave the following results:

TABLE V

COMPARATIVE PROTEIN MODIFICATION OF AMERICAN MALTS

Type of malt	6-Row Manchuria			California	2-Row	
Malt number	1	2	3	4	5	6
Malt N, %	2.085	2.203	2.223	2.040	1.617	1.608
	Basis cold water extract					
SN, %	0.475	0.504	0.536	0.469		
SN in % of N	22.8	22.9	24.1	23.0		
PSN, %	0.343	0.380	0.408	0.314		
PSN in % of N	16.5	17.3	18.4	15.4		
FN, %	0.097	0.133	0.128	0.102		
FN in % of N	4.7	6.0	5.8	5.0		
FN in % of SN	20.4	26.4	23.9	21.8		
FN in % of PSN	28.3	35.0	31.4	32.5		
	Basis laboratory wort					
SN, %	0.686	0.780	0.770	0.635	0.578	0.628
SN in % of N (DM)	32.9	35.4	34.6	31.1	35.8	39.1
PSN, %	0.667	0.725	0.743	0.607	0.551	0.338
PSN in % of N	32.0	32.9	33.4	29.7	34.1	33.5
FN, %		0.211	0.159	0.167	0.174	0.224
FN in % of N		9.6	7.2	8.2	10.8	13.9
FN in % of SN		27.1	20.7	26.3	30.1	35.7
FN in % of PSN		29.1	21.4	27.5	31.6	41.6

In addition, four more malts of the Manchuria type were examined to determine the relationship between soluble nitrogen of the wort and total malt nitrogen.

Malt number	7	8	9	10
Malt N, %	2.132	2.221	2.327	2.446
SN of wort in % of malt N	35.2	35.8	33.1	32.5

Although only a limited number of malts were examined, these analyses reveal some interesting characteristics of American malts as compared with foreign malts. The two samples of two-rowed malts show properties which are almost identical to similar European varieties. There is, however, a difference between the two samples in their degree of modification, which manifests itself in variation of soluble nitrogen expressed in percentage of malt nitrogen, and is still more pronounced in the formol nitrogen fractions, while their permanently soluble nitrogen values are very close. This goes to show how important it is to determine the several nitrogen fractions, for judgment on the basis of permanently soluble nitrogen alone would have been misleading.

With reference to the six-rowed varieties, it will be noted that the relative figures for soluble and permanently soluble nitrogen and the absolute and relative values for formol nitrogen, in general, are considerably lower in both cold water extract and wort than the corresponding values reported for two-rowed varieties. Due to the high nitrogen content of our malts this is not surprising. All the indications point to the fact that most of the malts are well modified for American brewing conditions. An exception is noted in malt No. 3 which, although normal in all aspects of the cold water extract and in soluble and permanently soluble nitrogen of the wort, showed a low formol nitrogen fraction in the wort. This apparently was due to low proteolytic activity. Likewise, the California malt No. 4, while normal in formol nitrogen, discloses somewhat low soluble and permanently soluble nitrogen values. Both malts presented difficulties in brewing operations and required readjustment of the brewing procedure.

The data presented herewith are too limited to draw any conclusions. Examination of a great number of American barley and malt samples of various origins and seasons, including observations of their behavior during the malting and brewing processes, is necessary before the usefulness of the nitrogen methods disclosed herewith for evaluation of malt intended for brewing definitely can be established. The present discussion purports only to indicate the possibilities of applying these new criteria to the study of American malts. This obviously does ex-

clude the necessity for fundamental research into the nature of barley and malt constituents and their relationship to beer quality.

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THE EFFECT ON LOAF VOLUME, OF PROVING DOUGHS TO A DEFINITE HEIGHT AS COMPARED WITH FOR A FIXED TIME[1,2]

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According to the specifications of the Official Baking Test of the American Association of Cereal Chemists (Geddes, 1934), a fixed proving time of 55 minutes is employed regardless of the type of flour under investigation. Also, whenever supplementary formulas are introduced, the same proving procedure is followed. This was shown by the results of the survey of test baking procedures employed in America, conducted by Aitken (1934), when only a comparatively small percentage of those who furnished information reported a change in proving technique with variable baking formulas. It is apparently assumed that all types of flour have the same optimum proving time and that ingredients added to stimulate yeast activity, assist gluten development and enhance gas production, exert little or no influence upon the proving rate of the dough. In addition, it is generally recognized that different bakers do not exert the same degree of pressure in molding, hence the height between the surface of the dough and the top of the pan must vary.

Under these conditions, replicate doughs fail to register exactly the same height after the same proving time and differences in loaf volume might be materially reduced by proving to a definite height. While the selection of a suitable height would be quite as arbitrary as employing a fixed time, the experiment here reported was undertaken primarily to determine which of the two proving procedures gave the most satisfactory results when corresponding flours were baked in replicate by the same and by different bakers.

Experimental

For the purpose of this study, two flours commercially milled from Canadian hard red spring wheat were chosen—one a short patent and the other a second clear—the protein, ash, and diastatic activity values for which are recorded in Table I.

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[1] Published as paper No. 107 of the Associate Committee on Grain Research, National Research Council of Canada and Dominion Department of Agriculture.

[2] Subcommittee report, 1935–36 Committee on the Standardization of Laboratory Baking.

TABLE I

MISCELLANEOUS DATA ON THE TWO FLOURS

Sample No.	Flour	Moisture	13.5% Moisture basis		
			Protein	Ash	Diastatic activity
		%	($N \times 5.7$) %	%	(*mg. maltose per 10 g. flour*)
1	Commercial short patent (bleached and matured)	14.0	12.5	0.39	222
2	Commercial second clear (bleached and matured)	13.3	15.1	1.06	268

The baking tests were conducted by two operators employing two formulas, *i.e.*, the malt-phosphate-bromate (0.3% diastatic malt—250° Lintner, 0.1% $NH_4H_2PO_4$, and 0.001% $KBrO_3$), and the bromate (0.001% $KBrO_3$), the other dough ingredients and baking procedure being as specified in the A. A. C. C. baking test (excepting when the dough was proved to a definite height), using low-form tins and a Hobart mixer. The proving height selected was 8.8 cm., measured from the top of the shelf upon which the pan rests to the centre of the surface of the dough, the device described by Aitken (1930) being employed to indicate when this height had been reached. The height chosen corresponds to that registered by a dough made from experimentally milled No. 1 Northern flour when proved 55 minutes, using the malt-phosphate-bromate procedure.

To obtain information regarding the effect of proving to height and proving to time on variability of replicates, with the same and between different bakers and also the effect of baking formula, each of the two operators baked five replicates from each flour employing both proving procedures, using the malt-phosphate-bromate baking method on one day and the bromate method on another. On both days the samples were randomized both for proving methods and operators.

As an indication of the variation in height between corresponding doughs when proved by both methods, the height of the dough in the pan was recorded just prior to transference to the oven.

The maximum, minimum, and mean loaf volumes, mean proving times, and dough heights, together with the results of gas production tests obtained by the manometric method of Blish, Sandstedt, and Astleford (1932), are recorded in Table II. In order to segregate and test the significance of variations traceable to specific sources, the loaf

TABLE II

LOAF VOLUMES, PROVING TIMES, AND DOUGH HEIGHTS

Baking formula	Baker No. 1				Baker No. 2			
	Patent flour		Clear flour		Patent flour		Clear flour	
	Height	Time	Height	Time	Height	Time	Height	Time
Loaf Volumes in Cubic Centimetres								
Malt-phosphate-bromate								
Mean	718	715	705	746	724	738	708	763
Min.	690	690	675	720	710	705	690	750
Max.	730	740	720	770	735	760	720	790
Bromate								
Mean	714	662	678	683	749	641	682	685
Min.	700	635	670	670	705	620	660	680
Max.	750	690	690	695	780	685	705	700
Mean Proving Times in Minutes								
Malt-phosphate-bromate	55	55	47	55	56	55	46	55
Bromate	82	55	53	55	82	55	51	55
Mean Dough Heights in Centimetres								
Malt-phosphate-bromate	8.7	8.8	8.7	9.3	8.7	8.6	8.7	9.2
Bromate	8.6	7.8	8.7	9.0	8.6	7.5	8.7	9.0
Gas Production in Millimetres Mercury Pressure during Proving Period								
Malt-phosphate-bromate	109	109	115	136				
Bromate	164	97	123	136				

volume data were analyzed by the method of variance developed by Fisher (1934). The results, which are shown in Table III, are expressed as F values with their corresponding 5% points, as recently outlined by Snedecor (1934).

Considering the loaf volume data in Table II, it will be noted that with the malt-phosphate-bromate formula, both operators obtained similar loaf volumes with the patent flour by both proving methods, but with the clear flour the time-proved loaves were the larger. With the bromate baking formula, in which no provision is made for supplementing gassing power, the loaf volumes by both operators were almost iden-

tical with both proving procedures for the clear flour, but the height-proved volumes were definitely larger for the patent flour.

TABLE III

ANALYSES OF VARIANCE—BOTH BAKERS COMBINED

Variance due to:	Malt-phosphate-bromate formula					
	Proving to height			Proving to time		
	F	5% pt.	S.E.	F	5% pt.	S.E.
Between flours	30.22	4.75		72.65	4.75	
Between bakers	2.91	4.75		3.71	4.75	
Between replicates	22.37	3.26		27.25	3.26	
Interaction—Flours × Bakers	.32	4.75		34.19	4.75	
Error			5.90 cc.			7.35 cc.
	Bromate formula					
Between flours	27.17	4.75		22.53	4.75	
Between bakers	3.90	4.75		1.92	4.75	
Between replicates	.76	3.26		2.47	3.26	
Interaction—Flours × Bakers	2.46	4.75		2.82	4.75	
Error			22.09 cc.			15.31 cc.

By both baking formulas and with both operators, the height-proved doughs placed the patent before the clear on the basis of flour strength as indicated by loaf volume, while with the time-proved doughs the order was reversed; in no case did any dough have an overproved appearance.

This anomalous situation may be due either to the fact that a proving time of 55 minutes is too short for flours of the "patent" type, or that the proving height employed was too low for flours of the "clear" type. In this connection, the proving times and dough heights recorded in Table II are of interest. With the malt-phosphate-bromate formula the patent flour gave approximately equal proving times and dough heights by the two proving procedures but the clear flour required a shorter proving time and gave a lower dough height by the "height" proving procedure. On the other hand, with the bromate formula, the patent flour required a longer proving time and gave a greater dough height by the proving-to-height procedure, while the clear flour gave approximately equal times and heights for the two proving procedures.

A comparison of these data with those for loaf volume shows a close relation; for each flour and baking formula the relative volumes obtained by the two proving methods are in the same order as the height of the doughs at the time of going to the oven. Moreover, proving to height considerably reduces the differences in loaf volume obtained by the two baking formulas; in fact for the patent flour the results are practically identical. From the F values and their 5% points given in Table III it will be noted that proving to height gives better differentiation between flours with the bromate formula, and proving to time with the malt-phosphate-bromate.

These observations suggest that the differences in proving time, dough height and loaf volume for the two proving procedures are associated with variations in gas production in the doughs, but actual determinations of the gas produced during the different proving times required to bring corresponding doughs to the same height, showed wide differences.

The variance analyses given in Table III show that while there were no significant differences between bakers for either baking formula or proving procedure, the results for the two proving methods in regard to differences "between flours," "between bakers," "between replicates," and the interaction of "Flours x Bakers" are entirely inconsistent for the two baking formulas.

Discussion

The limited series of experiments undertaken does not provide a clear-cut answer as to the relative merits of the two proving methods in regard to such factors as differentiation between flours and replicability within and between bakers, since opposing results were given by the two baking formulas. They show, however, that the height reached by the dough at the end of the proving period has an appreciable influence on the volume of the resulting loaf. The quantity of gas produced in the different times required to prove the doughs made from the two flours by the bromate and malt-phosphate-bromate formulas to the same height varied widely, indicating that the quantity of the gluten and rate and extent of its modification are contributory factors. Accordingly, if adjustments were made in the baking formula to equalize the gas production of doughs during proving, these would not result in a fixed proving time giving identical results with those obtained by proving to a definite height. Under such conditions, the latter method would tend to overcome variations in gluten quantity and the extent of its development as a result of fermentation; these are the very factors which it is desired should be reflected in the final loaf, and hence of the two methods, proving for a fixed time appears to be preferable.

From the practical standpoint, proving to a definite height would materially reduce the number of loaves which can be baked daily. The continuous procedure which follows with the standard time method is not applicable when doughs are proved to height, due to the variations in proving rate of different flour doughs. Also provision has to be made for longer intervals between the mixing of the doughs to prevent over-crowding of the oven at any one time. In addition to this, more than one baker is required if a large number of loaves are to be baked on any one day, since doughs are being transferred to and taken from the oven at irregular intervals, which materially interferes with the routine of the baking.

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THE RELATIVE LOSS IN PIGMENT CONTENT OF DURUM WHEAT, SEMOLINA AND SPAGHETTI STORED UNDER VARIOUS CONDITIONS [1]

D. S. BINNINGTON [2] and W. F. GEDDES [3]

(Read at the Annual Meeting, June 1936)

Introduction

Intensity of yellow pigmentation is generally considered to be a factor of major importance in durum wheats and semolinas employed in the manufacture of macaroni products. The pigments of durum wheat and semolina may be broadly classified into two groups: *Carotenoids* such as carotene and xanthophyll, and *Flavones*. The susceptibility of carotenoid pigments to oxidation with consequent loss of color is well known, and this results in a gradual bleaching of the sample with age. With wheats and flours intended for baking purposes, this color loss is a desirable condition, but with durum wheat and semolina the reverse is the case. Under these circumstances, some knowledge of the magnitude of such changes taking place under varied storage conditions is highly desirable, particularly in experimental work where samples may have to be stored for some time prior to testing. The following study was therefore conducted in order to obtain information on this point.

Experimental

The materials employed in this study were derived from a large sample of 1934 crop No. 1 C. W. Amber Durum wheat obtained from the Winnipeg Inspection Office and constituting a representative average of the grade for the crop year. The wheat was thoroughly cleaned and composited, and a portion milled into semolina using the experimental technique described by Binnington and Geddes.[4] A portion of the semolina was then processed into spaghetti, employing the technique also described by these authors. Analytical values for these samples are presented in Table I.

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[1] Contribution from the Grain Research Laboratory, Board of Grain Commissioners, Winnipeg, Manitoba, with financial assistance from the National Research Council of Canada. Published as paper No. 103 of the Associate Committee on Grain Research, National Research Council of Canada and Dominion Department of Agriculture.

[2] Research Assistant, Associate Committee on Grain Research.

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[4] Binnington, D. S., and Geddes, W. F. Experimental durum milling and macaroni making technique. Cereal Chem. 13: 497–521 (1936).

TABLE I

PROTEIN, MOISTURE, ASH, AND CAROTENE CONTENTS OF WHEAT, SEMOLINA AND SPAGHETTI

Sample	Protein[1]	Moisture	Ash[1]	Carotene[1]
	%	%	%	*p.p.m.*
Wheat	13.4	11.04	1.34	3.86
Semolina	11.6	13.08	0.55	2.91
Spaghetti	—	9.10	—	0.52

[1] Results expressed on a 13.5% moisture basis.

It will be noted that a very considerable drop in pigment content exists between the semolina and the spaghetti produced therefrom. This point has been investigated at some length and appears to be partly associated with a change in color of the flavone pigments which exhibit an indicator action, and partly to adsorption of, or change in the conditions of association of the carotenoids. That this loss of pigment is only apparent can be proved by the use of certain solvents which in the majority of cases yield values closely approximating the original for the semolina.

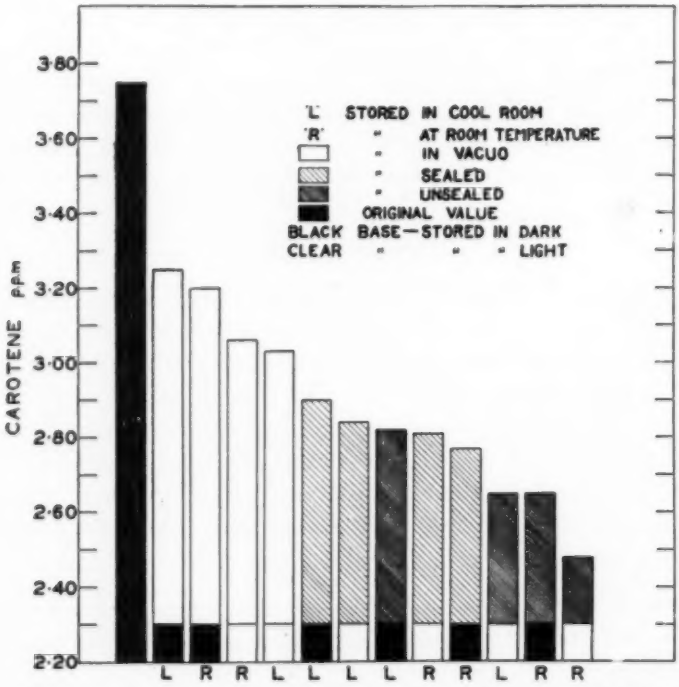

Figure 1. Histograms showing changes in the carotene content of durum wheat after twelve months' storage under various conditions.

Carotenoid pigment contents were determined by the official A. A. C. C. spectrophotometric procedure, the transmittancies being read with a Bausch and Lomb Universal Spectrophotometer. Naph-

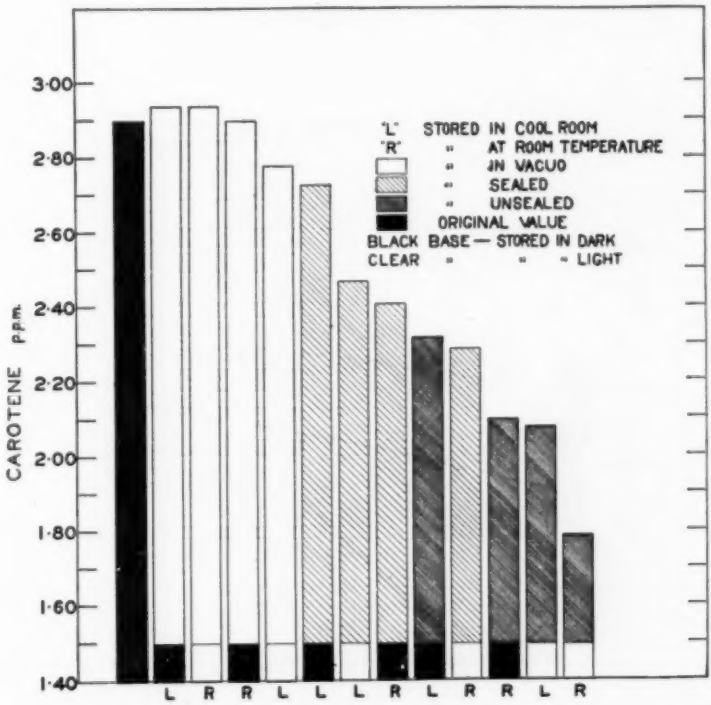

Figure 2. Histograms showing changes in the carotene content of semolina after twelve months' storage under various conditions.

tha-alcohol solvent (93 : 7) as described by Geddes, Binnington, and Whiteside [5] was used in all cases, the samples being prepared by grinding in a Wiley Mill fitted with a 1/2 mm. screen. Moistures were determined at the same time as the pigment analyses and the values corrected to a 13.5% moisture basis.

The samples were stored at two temperature levels, (1) average room (70–85° F.) and (2) cool room (40–60° F.); also within each of these temperature levels in (*a*) dark, and (*b*) light (not direct sunlight). Each set of samples stored under the above conditions was sealed as follows: (1) free access of air (containers plugged with absorbent cotton), (2) friction top cans and corked bottles, and (3) evacuated glass tubes. Pigment determinations were conducted after 3, 6, and 12 months.

[5] Geddes, W. F., Binnington, D. S., and Whiteside, A. G. O. A simplified method for the determination of carotene in flour extracts. Cereal Chem. 11: 1–24 (1934).

The results obtained are presented in the form of histograms in Figures 1, 2, and 3. For the sake of brevity and ease of comparison, only the original and final values are given. In order to secure an

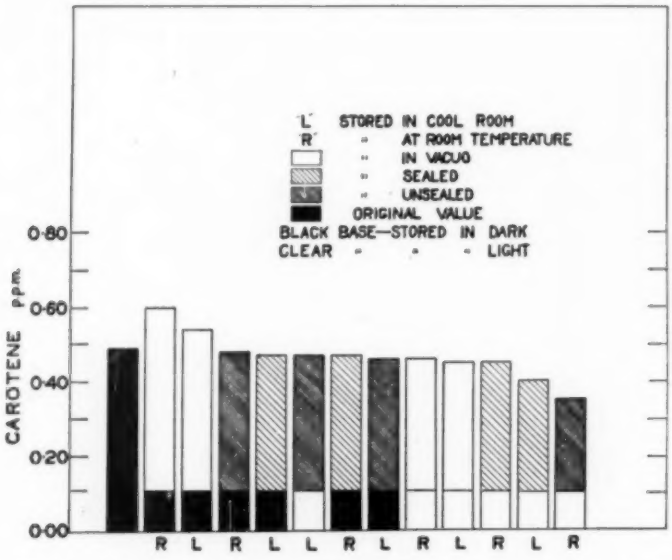

Figure 3. Histograms showing changes in the carotene content of spaghetti after twelve months' storage under various conditions.

idea of the trends, however, the data for wheat and semolina have been averaged for all storage conditions at 3, 6, and 12 months, and these values are shown graphically in Figure 4. Spaghetti showed so little change that the results have not been graphed.

Discussion of Results

WHEAT. Vacuum storage showed the smallest loss of pigment, sealed containers next, and unsealed the most; low temperatures a smaller loss than room temperature, and dark storage less than daylight. Under practical storage conditions, *i.e.*, sealed containers kept in the dark at moderately low temperatures, a pigment loss of 0.85 p.p.m. (22.7%) was found in one year. With the same conditions, but at room temperature, the loss amounted to 0.98 p.p.m. or 26.1%.

SEMOLINA. The order of loss in regard to the various storage conditions employed was similar to that found with wheat. Sealed containers in the dark at low temperature lost 0.17 p.p.m. (5.9%), and under the same conditions but at room temperature the loss amounted to 0.49 p.p.m. or 16.9%.

SPAGHETTI. Considerable irregularity was found to exist within the individual tests of this series and in the early stages many samples showed an apparent slight but definite *increase* in pigment content.

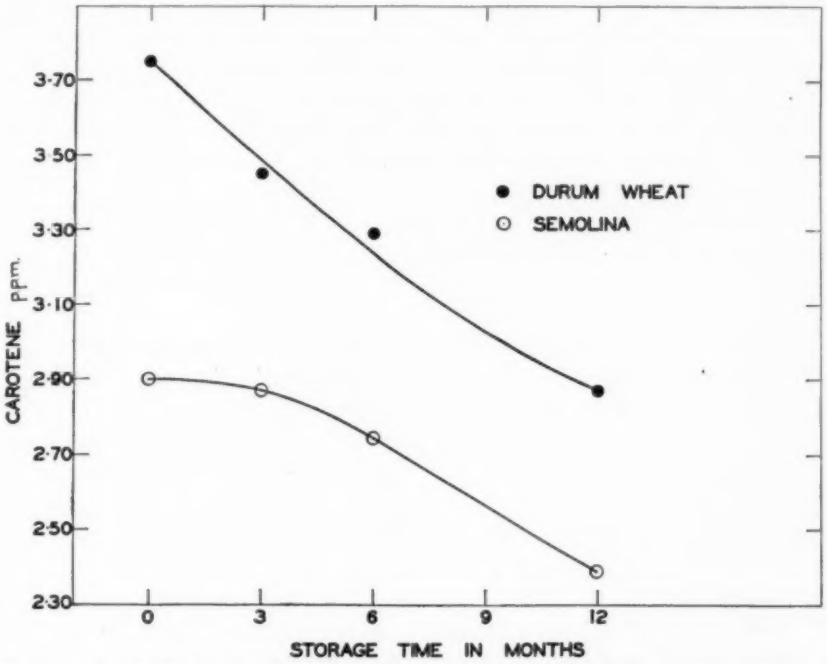

Figure 4. Graphs showing the changes (means of all storage conditions) in the carotene content of durum wheat and semolina with increasing time of storage.

This may be due to a partial reversal of the conditions previously discussed, which produce such a decided apparent drop in pigment content during processing. In any case the total loss is very much smaller than with either wheat or semolina, and with the exception of sealed samples stored at low temperature in the light, and unsealed samples stored at room temperature in the light, the final values are practically identical and show no significant decrease at the end of a year's storage.

The results in general follow the trends to be expected with the storage conditions employed. An interesting point, however, is the fact that the losses for semolina are decidedly lower than for wheat under corresponding storage conditions, despite the fact that the semolina possessed a definitely higher initial moisture content than the wheat. It would appear from these results that experimental material which has to be stored for any length of time should be milled upon receipt and stored as semolina at as low a temperature as possible in well sealed cans in order to minimize pigment loss.

Summary

Samples of spaghetti and semolina experimentally processed from durum wheat, together with the wheat itself, have been stored under a variety of conditions for a period of one year.

Pigment determinations have been made and data are presented showing the general trends of pigment change during this period.

The conditions of storage in order of increasing loss of pigment were: vacuuo, sealed containers, and unsealed containers. The amount of loss also increased from low to higher temperatures and was greater in daylight than in the dark.

At the end of one year's storage, spaghetti underwent little change, semolina next and wheat the most.

AN INVESTIGATION INTO THE CAUSE OF THE DEGRADING EFFECT OF WHEAT GERM ON THE BAKING QUALITY OF FLOUR

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Baking quality in flour is largely dependent on the quality of the gluten, which is further affected by the presence of various substances in the dough. Though investigations have been few, the harmful effect of wheat germ on the baking quality of flour has generally been ascribed to the presence of lipoids, but it is conceivable that the poor baking quality of wheat germ is due to some property inherent also in weak flours, where in many cases it has been shown that the poor quality was due to substances soluble either in ether or water.

Sullivan and Near (1927) considered that the poor quality of the gluten from the whole wheat as compared with that from a patent flour was due to a higher lipoid content. Johnson (1928) suggested that flour might be improved by extracting with ether. He found that in every case the bread produced from the ether-extracted flour was superior to that from the corresponding natural flour, the greatest improvement being found in flours richest in lipoids. On the other hand, Martin and Whitcomb (1932) noted that only in some cases was there an improvement in the loaves of ether-extracted flours, other flours after extraction showing a deleterious effect. They concluded that the quality

[1] The author wishes to express her thanks to the New Zealand Wheat Research Institute for baking and scoring test loaves.

of the gluten was probably determined " more by difference in the chemical constitution of the gluten proteins and their physical-chemical environment than by the presence or absence of fat-like substances."

In the present study wheat germ was extracted with ether and baking tests made including in the flour ether-extracted germ. Table I gives the results obtained. The points allowed in scoring the loaves were—general appearance, 8; bloom, 8; flavour, 10; texture, 10; crumb colour, 8; and pile, 6; giving a total score of 50 points.

TABLE I

Effect of the Addition of Wheat Germ on the Baking Quality of Flour

Additions to flour	Score		
	General appearance	Texture	Total
Standard flour	7	7	40
Standard flour + 2% pure germ	5	4	22
Standard flour + 2% fat-extracted germ	2	2	10
Standard flour + 2% dried fat extracted germ	3	2	12

With the ether extract removed no improvement was apparent in the loaves, the germ characteristics still being most pronounced. From this it was assumed that the ether extract, that is the fats, is not responsible for the harmful effects shown by the wheat germ. To prove this conclusively, the oil extracted was incorporated into a loaf. One cc. of oil added, corresponding to 10 g. of germ or 8% of the total flour, produced no difference in the loaf, while 5 cc. of the oil showed no appreciable difference as can be seen from Table II and from Figure 1.

TABLE II

Effect of the Addition of Wheat Germ Oil on the Baking Quality of Flour

Additions to flour	Score		
	General appearance	Texture	Total
Standard flour	7	7	38
Standard flour + 1 cc. of germ oil	7	7	38
Standard flour + 5 cc. of germ oil	7	7	37

Since the addition of ether-extracted germ to flour still produced degrading effects it was probable that some other solvent would remove the harmful substances. Sharp and Gortner (1923) had improved the baking quality of flour by extracting with distilled water, thereby re-

moving the water-soluble ash salts. Working (1924) extended this work investigating the removal of lipoids and other water-soluble substances and showed by baking tests, that as the result of his treatments low grade flours became almost equal in strength to patent flours. However, Johnson (*loc. cit.*) found that treatment of flour with water had little effect on the bread-making properties of flour.

Since wheat germ is not improved by ether extraction, germ which had already been ether extracted was then extracted with (1) absolute alcohol, (2) water, and baking tests made on flour with the residues. Extraction with alcohol made no difference in the effect of the germ on the loaf, while inclusion of the water-extracted residue gave an improvement in the loaf over the raw germ. It was therefore concluded that the harmful substances were insoluble in ether and alcohol, but soluble in water. Further baking tests were performed and it was clearly shown that the water-extracted residue had no effect on the baking quality, while the water extract itself had marked degrading effects but at the same time was slightly better than the raw germ, so that the water extract possessed a large proportion of the degrading substance. Also, the water extracts of fat-extracted and pure germ had about equal degrading effects, but by heating and removing the coagulated portion of the water extract a slight improvement was brought about. Results are given in Table III, and loaves are shown in Figure 1.

TABLE III

THE EFFECT OF WATER EXTRACTS OF WHEAT GERM ON THE BAKING QUALITY OF FLOUR

Additions to flour	Score		
	Texture	Crumb colour	Total
Standard flour	7	6	35
Standard flour + wet freshly extracted germ	5	7	36
Standard flour + water extract	4	2	24
Standard flour + water extract with coaguable portion removed	7	5	34

Working (*loc. cit.*) dialyzed a series of flours which showed a definite improvement in quality over those merely digested in water, while flours digested and then extracted with water showed still greater improvement due to the removal of water-soluble substances as well as ash salts. He suggested that besides phosphatides, water-soluble proteins and even pentosans may be important factors deteriorating gluten. Now in the water extract from wheat germ there will be present besides water-soluble ash salts and a small amount of lipoid, water-soluble proteins, non-protein nitrogen compounds, and other organic compounds.

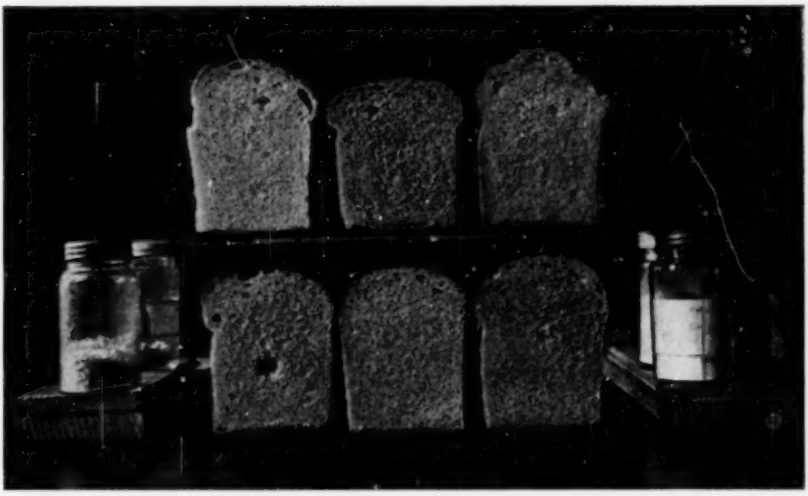

Fig. 1. Loaves showing the effect of wheat germ, wheat germ oil and water extracts of wheat germ on the baking quality of flour. *Top row*—control, flour + wheat germ, flour + 5 cc. wheat germ oil. *Bottom row*—flour + water extract, flour + residue from soaking, flour + boiled and strained extract.

By dialyzing, some of these substances may be removed. The water extract from 80 g. of wheat germ extracted with a litre of water for 24 hours at room temperature was dialyzed for 3 days. This dialyzed solution was found, in a baking test, to have lost all the germ characteristics; the loaf produced being equal to, or better than, the standard. In this case also, removal of the coaguable portion by heating produced a slightly better loaf. The conclusion was drawn that the constituents of the wheat germ giving degraded baking qualities are water soluble and dialyzable. Results are given in Table IV.

TABLE IV

THE EFFECT OF DIALYZED WATER EXTRACT OF WHEAT GERM ON THE BAKING QUALITY OF FLOUR

Additions to flour	Score			
	Texture	General appearance	Flavour	Total
Standard flour	7	6	7	36
Standard flour + water extract	2	3	1	12
Standard flour + water extract with coaguable portion removed	4	4	3	20
Standard flour + dialyzed water extract	5	8	5	31
Standard flour + dialyzed water extract with coaguable portion removed	7	7	7	38

It was noticed that the water extract during dialysis deposited a fine white precipitate. A clear solution of water extract after standing for

4 days formed a similar precipitate. In order to show that a time factor was not responsible for the improvement by dialysis, in which case improvement might be caused by the decomposition of some organic substance present, a baking test was made including water extract of germ which had been kept for 4 days. This loaf still showed all the germ characteristics as shown in Table V.

TABLE V

EFFECT OF WATER-EXTRACT OF GERM ON BAKING QUALITY

Additions to flour	Score			
	Flavour	Texture	Crumb colour	Total
Standard flour	6	6	5.5	32.5
Standard flour + water extract of germ left standing 4 days	1	3	1	15
Standard flour + water extract dialyzed for 2 days	3	3	4	27

The possibility arises that the flour used might be favourable to the dialyzed solution. To test this a wide variety of flours was experimented with. To each flour there were added: pure germ (A), water extract of germ (B), water extract with the coaguable material removed (C), dialyzed water extract (D), dialyzed water extract with the coaguable material removed (E). Results are given in Tables VIa, b, c, and d. 2½ g. of wheat germ, or 2% of the total flour was added, and this is denoted in Table VI by A. The extracts of germ as given above corresponding with this amount, 2%, are denoted by B, C, D, and E.

TABLE VIa

THE EFFECT OF WHEAT GERM, WATER EXTRACT, AND DIALYZED WATER EXTRACT OF WHEAT GERM ON GENERAL APPEARANCE

Flour type	Flour control	Additions to flour				
		Flour + A	Flour + B	Flour + C	Flour + D	Flour + E
N. 4	7	6	7	6	7	7
D.H.B.	6	6	6	6	7	6
Woods 1	6	5	5	5	5	7
Woods 2	5	4	5	3	5	6
H. & T.	6	7	6	7	6	7
Ireland	7	3	6	5	6	–
Waikari D29	6	–	3	4	8	7

TABLE VIb

THE EFFECT OF WHEAT GERM, WATER EXTRACT, AND DIALYZED WATER EXTRACT OF WHEAT GERM ON LOAF TEXTURE

Flour type	Flour control	Additions to flour				
		Flour + A	Flour + B	Flour + C	Flour + D	Flour + E
N. 4	7	5	5	6	6	7
D.H.B.	7	6	5	5	5	6
Woods 1	7	4	4	4	4	7
Woods 2	6	4	4	4	4	6
H. & T.	6	6	7	7	6	6
Ireland	7	3	6	6	7	–
Waikari D29	7	–	2	4	5	7

TABLE VIc

THE EFFECT OF WHEAT GERM, WATER EXTRACT, AND DIALYZED WATER EXTRACT OF WHEAT GERM ON CRUMB COLOUR

Flour type	Flour control	Additions to flour				
		Flour + A	Flour + B	Flour + C	Flour + D	Flour + E
N. 4	7	5	4	7	5	7
D.H.B.	7	4	4	3	4	5
Woods 1	7	4	2	3	3	6
Woods 2	5	2	2	3	3	6
H. & T.	6	6	6	6	5	6
Ireland	7	4	6	6	6	–
Waikari D29	7	–	2	3	5	6

TABLE VId

EFFECT OF WHEAT GERM, WATER EXTRACT, AND DIALYZED WATER EXTRACT OF WHEAT GERM ON BAKING QUALITY

Flour type	Flour control	Additions to flour				
		Flour + A	Flour + B	Flour + C	Flour + D	Flour + E
N. 4	38	30	30	36	33	40
D.H.B.	38	29	27	28	30	34
Woods 1	37	24	20	23	22	29
Woods 2	32	20	20	19	22	35
H. & T.	35	33	34	37	34	35
Ireland	40	21	33	33	36	—
Waikari D29	36	—	12	20	31	38

From these results it will be seen that no matter what the type of flour, the water extract of germ has a degrading effect, while the dialyzed water extract no longer shows these properties, and, in fact, produces an improvement in the loaf. This radical change brought about by dialysis suggests that the minerals and any organic bases, acids,

or amido compounds present which could be removed by dialyzing may be responsible for the deleterious effects. The solutions were therefore analyzed for their mineral constituents.

Analysis of Water Extract and Dialyzed Water Extract

The ash from the water extract was invariably dark and partially fused while the dialyzed water extract gave a light coloured fluffy ash. Johnson and Scott (1928) state that flour ashes of high potassium content and low phosphorous content have fluffy ashes, while ones having low potassium content and high phosphorous content tend to be fused. This, as will be seen from results obtained, does not occur with ashes from water and dialyzed water extracts of germ.

On a dry-matter basis the ash from the water extract was 3.77% of the original germ, which after dialyzing for 2 days dropped to 1.85% and on the third day to 0.895%. Analysis of the twice dialyzed solution for its inorganic constituents was made on the 3 days' dialyzed solution. *Iron, aluminium, calcium, and magnesium* were determined on the same weighed ash sample. Iron and aluminium were precipitated together as phosphates, the iron then determined volumetrically and in the same solution the phosphoric acid was determined. Calcium was precipitated as the oxalate and finally weighed as the oxide, and magnesium was determined in the form of magnesium pyrophosphate. *Chlorine* was determined on a separate ash sample and precipitated as silver chloride. *Sulphur* was determined on both ashed and water samples, being collected as barium sulphate. *Potassium and sodium* were determined after removal of the other metals, using the A.O.A.C. method for plants, and separating them by the perchloric method.

TABLE VII

Ash Analysis of Mineral Constituents Expressed as Per Cent Ash

	P_2O_5	SO_3	K_2O	Na_2O	CaO	MgO	Fe_2O_3	Al_2O_3	Cl
Water extract	36.06	7.16	45.54	4.08	1.3	7.58	0.56	0.35	0.054
Dialyzed water extract	60.78	4.43	3.04	4.74	13.28	13.56	0.68	—	—

Results are given in Tables VII and VIII from which it will be seen that the greatest portion of the ash is present in the form of a phosphate. Dialyzing brings about a decrease first in the monovalent elements so that the percentage of Ca, Mg, and Fe in the ash from the dialyzed solution is higher than that from the water extract.

Teller (1896) has shown that the ratio CaO/MgO in the ash of flours increases in value from low grade flours to fine flours. In the

water extract of wheat germ this ratio is 0.17, but after dialysis it rises to 0.97 indicating that an improved loaf will be produced.

TABLE VIII

MINERAL CONSTITUENTS EXPRESSED AS PER CENT DRY GERM

	P_2O_5	SO_3	K_2O	Na_2O	CaO	MgO	Fe_2O_3	Al_2O_3	Cl
Water extract	1.36	0.27	1.72	0.154	0.049	0.286	0.021	0.013	0.002
Dialyzed extract	0.544	0.0397	0.027	0.042	0.119	0.121	0.03	—	—

The nitrogen content of the water extract of wheat germ changes on dialysis from 2.55% to 1.62%, a change of 36.48% of the total nitrogen. Proteins being of a colloidal nature would not be removed by dialysis but non-protein nitrogen compounds could pass through a parchment membrane. Teller and Teller (1932) separated out the various protein groups from wheat bran and their methods were used to separate the nitrogen groups in wheat germ. Besides the non-protein nitrogen, albumins were the only other protein group present. In Table IX are given the amounts of the nitrogen compounds present in the water and dialyzed water extracts from which it is apparent that the loss occurring in the non-protein nitrogen compounds corresponds with the loss of nitrogen brought about by dialyzing.

TABLE IX

NITROGEN OF DIFFERENT COMPOUNDS PRESENT IN WATER EXTRACT AND DIALYZED WATER EXTRACT OF GERM

	Total nitrogen	Albumin	Non-protein
Water extract	2.55	0.78	1.78
Dialyzed water extract	1.62	0.74	0.85

To replace in the dough the inorganic salts of phosphoric acid formed by the hydrolysis of phytin on water extraction, in the same combination as they existed in the germ, would be impossible, but the effect of various nonprotein compounds on the baking quality could be tested. Teller (1932) enumerates the nonprotein compounds which have been found in wheat. Of these only three—asparagine, betain, and lecithin—were obtainable for the work under discussion.

Separation of Asparagine from Wheat Germ

A small amount of asparagine was separated from wheat germ using the method given by Onslow (1923, p. 150).

Effect of Non-protein Nitrogen Compounds on Baking Quality

To simulate the effect of these substances, there was added, as well as the dialyzed solution, 0.236 g. of asparagine to one loaf, 0.1438 g. of betain to another, and to a third 0.0182 g. of lecithin. Results are given in Table X from which it will be seen that neither betain nor lecithin showed degrading effects, but 0.2% asparagine added to the flour produced a loaf showing properties similar to those given by the addition of wheat germ to flour. It is probable that further investigation would show that a smaller amount of asparagine would have the same effect and that other non-protein substances act similarly. Loaves are shown in Figure 2.

Fig. 2. Loaves showing the effect of added wheat germ, dialyzed extract and asparagine on baking quality. (1) Standard flour, (2) Flour + wheat germ, (3) Flour + dialyzed water extract, (4) Flour + asparagine.

TABLE X

Effect of Non-protein Nitrogen Compounds on Baking Quality

Additions to flour	Score		
	Appearance	Texture	Total
Standard flour	6	6	33
Standard flour + dialyzed solution	5	6	30
Standard flour + dialyzed solution + 0.236 g. asparagine	4	4	20
Standard flour + dialyzed solution + 0.1438 g. betain	5	6	28
Standard flour + dialyzed solution + 0.0182 g. lecithin	5	6	31

Working (*loc. cit.*) has suggested that phosphatide, the greatest percentage of which is located in the germ, lowers the baking quality

and he showed that the inclusion of wheat phosphatide or egg lecithin in flour produced a poor loaf. In this case the amount of lecithin introduced was much less than that used by Working and no harmful qualities were apparent. Geddes (1930) noted that wheat germ which had been previously heated before use showed no degrading effect and he suggested that the oxidation of the phosphatide produced the primary change. In order to ascertain the effect of heat on asparagine under similar conditions, wheat germ and 100 g. of wheat germ with 2 g. of asparagine were heated in sealed tins for 3 hours, and loaves baked containing 2% germ with and without added asparagine before and after heating. Results are given in Table XI which show that the asparagine after heating still has strong degrading effects, the improvement with heat treatment being due to a change in the germ, though this improvement is not given until a very high temperature is reached.

TABLE XI

THE EFFECT OF HEATING OF WHEAT GERM AND ASPARAGINE ON THE BAKING QUALITY OF FLOUR

	Score			
	Appearance	Texture	Pile	Total
Standard flour	6	7	4	34
Standard flour + wheat germ	3	3	1	16
Standard flour + heated wheat germ	7	4	2	28
Standard flour + wheat germ and asparagine	3	2	0	11
Standard flour + heated wheat germ and asparagine	4	2	1	16

Measurement of the pH of the Water Extract of Wheat Germ

That the pH of the water extract is responsible for the harmful qualities shown is unlikely for, as many workers have shown, the more acid solution under comparable conditions should produce the better loaf. Geddes (1930) states that acidulation of the dough does not reduce the harmful effects of the germ.

The pH of the water extract of wheat germ and the dialyzed water extract was measured by means of a Hellige Comparator using coloured glass standards. This method is not to be entirely relied on since a possible "protein" error would cause an increase in the pH of the solution. The water extract had a pH of 4.9; the dialyzed solution pH was 5.4.

The degrading effect of wheat germ has been attributed to the presence of oil weakening the gluten strands by lubrication, or else to the presence of coarse bran like substances diminishing the normal elasticity

of the gluten. In the present case where the water extract containing neither oil nor bran particles, produced the same result as wheat germ itself, the cause of the degrading effect must be looked for in water-soluble substances such as enzymes, soluble proteins, amino compounds, and inorganic salts, and it is highly improbable that only one substance will have this deleterious effect on the gluten quality.

Before the problem under discussion had fully developed, the wheat germ was submitted to a general analysis and the protein groups were separated out. Apparently the removal of heat coagulated albumins has a slight improving effect, but this was not further investigated. Though this work does not bear directly on the subject it has been included here as it may be of interest to other workers in this field.

General Analysis of Wheat Germ

Samples of wheat germ were submitted to a general analysis and results obtained unless otherwise stated are on a dry-matter basis.

Moisture was determined on 5 g. samples heated in a water oven at 100° for 5 hours, and varied according to the eight samples tested from 11.29% to 12.42%.

Fat was determined by extracting dried wheat germ with ether in a Soxhlet apparatus for 16 hours. The ether was previously purified by washing, standing over sodium hydroxide, distilling, and leaving it standing over sodium. Results varied according to samples from 10.42% to 14.50%. An undried sample had the value of 9.75%.

Nitrogen was determined on 2 g. samples using the Gunning-Arnold modification of the Kjeldahl method. Results varied from 5.39% to 5.44%, and using the factor 5.8 for conversion to crude protein results were from 31.25% to 31.54%.

Lipoid and lipoid phosphorous were estimated on 5 g. of the wheat germ sample using the method of the A.O.A.C. for cereal foods. The lipoid content of the undried pure germ was 10.50%, and for the dried solid residue from the water-extracted germ 16.08%, while the water extract contained 0.22%. Lipoid phosphorous in the undried pure germ, expressed as percentage lipoid, was 0.926%, expressed as per cent total dry germ 0.101%.

Phosphorous in all cases was determined by the gravimetric pyrophosphate method. In the undried pure germ it was 2.22%, in the dried pure germ 2.49%, in the undried ether-extracted germ 2.75%.

Ash was determined on 4.5 g. of wheat germ which was ignited in a silica crucible in an electric muffle furnace at 550° C. $\pm$ 5° C. until a constant weight was obtained. It was greyish in colour and tended to fuse slightly. Results varied according to samples from 4.63% to 5.20%.

Separation of Protein Groups from Wheat Germ

Teller and Teller (*loc. cit.*) separated out the wheat bran protein groups, and using their methods, a similar separation of nitrogen groups from pure wheat germ and fat-extracted wheat germ was made. Results are given in Tables XII and XIII.

TABLE XII

NITROGEN OF DIFFERENT COMPOUNDS FOUND IN WHEAT GERM AND FAT-EXTRACTED WHEAT GERM CALCULATED ON DRY-MATTER BASIS

	N compounds				
	Total N	Albumin	Globulin	Prolamine	Non-protein
Pure germ	4.73	1.30	0.85	0.74	1.82
Fat-extracted germ	5.60	2.34	1.11	0.67	1.45

TABLE XIII

NITROGEN OF DIFFERENT COMPOUNDS FOUND IN WHEAT GERM AND FAT-EXTRACTED WHEAT GERM CALCULATED AS PER CENT OF TOTAL NITROGEN

	N compounds			
	Albumin	Globulin	Prolamine	Non-protein
Pure germ	27.48	17.97	15.64	38.48
Fat-extracted germ	41.78	19.82	11.96	25.89

All nitrogen compounds were dissolved by 0.2% caustic soda.

Summary

Wheat germ after extraction with ether or/and alcohol does not possess improved baking qualities.

The water extract of wheat germ contains substances having a deleterious effect on the baking quality of flours. This may be slightly improved by removal of the coaguable portion by heating.

By dialyzing the water extract of wheat germ, substances harmful to the baking quality of flours are removed. The degrading effect is partly due to (1) the proportion of minerals present, in particular the amount of MgO relative to CaO; (2) the presence of the non-protein compound, asparagine. Wheat germ has been analysed for moisture, fat, nitrogen, phosphorous, lipoids, and ash content.

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A PRELIMINARY STUDY OF THE PHYSICAL SIGNIFICANCE OF CERTAIN PROPERTIES MEASURED BY THE CHOPIN EXTENSIMETER FOR TESTING FLOUR DOUGHS

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Introduction

In a number of recent publications a method has been described for measuring the viscosity (η) and rigidity modulus (n) of flour doughs (see Schofield and Scott Blair, 1932, 1933, 1933a, 1937). These properties are not independent of stress or strain, but it has been shown that if suitably standardized conditions are used, values of η and n (which can be expressed in absolute units) can be obtained which give direct information about the capacity of the dough to make good bread under English bakehouse conditions.

The most important properties of a dough for making English bread are—

Spring which is measured by the ratio of viscosity to a power of the modulus,

Extensibility which is normally associated with a relatively small fall in viscosity with rising stress, and

Tolerance which means that viscosity should not be too sensitive to changes in moisture content and fermentation-time.

This work was carried out jointly by the Physics Department, Rothamsted Experimental Station, Harpenden, England, and the Research Association of British Flour Millers, and is being published in a series of joint papers (Halton and Scott Blair, 1936, 1936a, 1937a).

Recent co-operation between these institutions and the Centre de Recherches Agronomiques, Versailles, France, has made possible an extension of the principles developed to a study of French conditions and a comparison with the Chopin extensimeter (Chopin, 1921, 1921a) now widely used in commercial flour and wheat testing throughout the continent of Europe.

In the Chopin technique, a bubble is blown in a thin strip of dough. The maximum pressure (P) and the square root of the volume attained before the bubble bursts (G) are automatically recorded. The air pressure required to cause the bubble to form is applied by means of a falling column of water, *i.e.*, at a regular but decreasing rate. The pressure in the bubble rises to a maximum, at which point the walls of the bubble are said to become permeable to air, although it continues to expand for some time longer before final rupture and collapse occur. Although the pressure passes through a maximum the surface of the bubble is still increasing, and there is no evidence that the stress per unit area does not continue to rise up to the final rupture. The viscosity of flour dough falls with rising stress (structural viscosity) but rises with increasing deformation (word-hardening (see Schofield and Scott Blair, 1932)). The variation of viscosity during the production of the bubble will thus be complex, but at the point of final rupture the viscosity may be defined as the momentary shearing stress divided by the rate of change of non-recoverable deformation. Since the rate at which air is supplied to the bubble is predetermined, it seems probable that the value of P should be correlated primarily with the viscosity of the dough.

Bakers use different quantities of water with different flours, so that the viscosities of doughs used in the bakehouse do not differ nearly as widely as do those of the Chopin test doughs which are all made up to contain the same quantity of water. Hence a flour of high water-absorbing capacity gives a dough of high viscosity when made up with the standard amount of water, whereas a flour of low absorption produces a dough of low viscosity. This suggests that under the standard conditions of the Chopin tests, P, being related to viscosity, should be a measure primarily of the water-absorbing capacity of the flour.

The significance of G is not so easy to predict, but the following line of argument is suggestive:

After the bursting of the bubble, the walls recover to about half their fully distended area. The total deformation is thus divisible into two parts, (*a*) recoverable deformation (σ_ϵ), and (*b*) non-recoverable (σ_η). σ_ϵ will be defined by the ratio of the shearing stress S to the shear modulus n. It therefore follows that the lower the modulus the bigger will be σ_ϵ for a given stress. Now the higher the viscosity the bigger will be the stress under the arbitrarily fixed rate of application of deformation (remembering that viscosity is defined as S/(rate of change of σ_η)) which will, in its turn, make for a bigger extension. The higher the viscosity, the greater the proportion of elastic to total deformation. It thus appears that a high η and a low n, which have

already been shown to give a big elastic recovery, are also the predominant factors in producing a high value of G (see Halton and Scott Blair, 1936). This, however, presupposes that the dough is reasonably extensible. It is clear that a "short" dough will tear before either a high pressure or extension can be produced. Now it has been shown that shortness is correlated with the rate at which η falls with rising stress (Halton and Scott Blair, 1936a), and some measure of extensibility is obtained from the ratio η_h/η_l (where η_l is viscosity at low stress (normal stress), and η_h at high stress).

It is appreciated that this argument is not strictly quantitative, but bearing this in mind, we can proceed to write a generalised equation of the type:

Equation (1) $$G = (f)\frac{\eta_l^a}{n^b}\cdot\frac{\eta_h^c}{\eta_l^d}$$ where a, b, c and d are unknown powers.

The baking value of flour has been shown to depend under English conditions, on suitable values of viscosity and shear modulus. The optimum relationship between these two properties, and the best conditions for their variation with other factors such as stress, age of dough, moisture content, *etc.*, have not yet been fully worked out; but although the requirements of the French market differ somewhat from the English, it is reasonable to suppose that whereas the relative importance of the different factors will not be the same, the same fundamental properties will be significant in both countries. In judging the potentialities of new varieties of wheat, it is not possible to do baking tests for lack of large enough samples, and the Chopin figures have to be taken as a criterion of value. It is thus of the greatest importance to find out what combination of physical properties the instrument really measures and a preliminary study of this problem forms the subject of the present note.

Experimental

Doughs made by the standard Chopin technique from some 35 flours of widely different origin were examined, measurements of P, G, η_h, η_l, and n being recorded. P and G were obtained from the Chopin diagram in the usual way. η_h was determined by measuring the rate of flow of a dough under a load[1] of about 80 to 85 Kg./cm.^{2} through a narrow brass tube of approximately 5 mm. diameter and 5 cm. length. No attempt was made to convert η_h into absolute units, and it is realised that, as a measure of true viscosity, it is only approximate. η_l and n were determined by the technique already described by Halton and Scott Blair (1936, 1936a) at a shearing stress[2] of 1200 dynes/cm.^{2}

[1] *i.e.*, a shearing stress of the order of 10^7 dynes/cm.^{2}.
[2] Since the technique is being described in an article in this journal (Cereal Chem., 14: 205), no further description of it will be given here.

In order to check that η_l/η_h was giving a measure of "shortness" as determined by feel, a spare portion of each dough was felt carefully before testing, and a note recorded. It was afterwards found that the mean values of η_l/η_h corresponding to those notes were as follows:

TABLE I

Note	Number of doughs	Mean η_l/η_h
Very short	8	28
Short	10	22
Short-medium	9	16
Medium	5	13
Medium-extensible	1	12
Extensible	1	9
Total	34	

This shows a good general agreement between shortness as assessed by feel and η_l/η_h.[3]

In order to test the validity of an equation of the general type of (1), still further simplifying assumptions must be made. As a first approximation it is assumed that $a = d$ and $c = 1$. Hence, we can write:

Equation (2) $$G = (f)\frac{\eta_h}{\eta^b}.$$

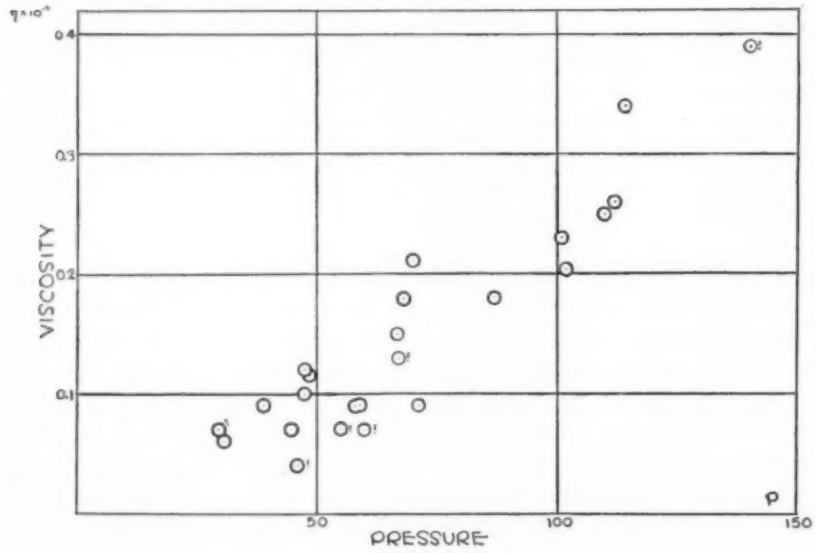

Fig. 1.

The data were examined to see whether any such correlation existed fitting different values for b, and it was found that the most satisfactory

[3] Since "shortness" does not have quite the same significance in different countries it should be stated that all assessments by feel were done by one of the authors (G.W.S.B.).

results were obtained when $b = 2.0$. This is interesting in view of the findings of Halton that η/n^x, which is a measure of spring, becomes independent of moisture content when x is given a value of slightly less than 2.0 for most flours. The results of the main experiment are shown in Figures 1 and 2. The general correlation between P and η_l

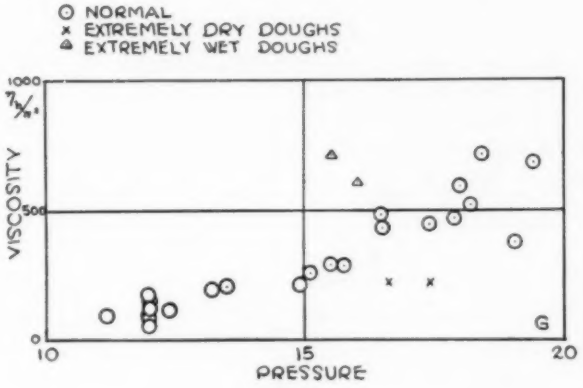

Fig. 2.

is clear from Figure 1. In Figure 2, values of G are plotted against η_h/n^2. The two samples marked (△) were exceptionally wet doughs, and the two marked (×) were abnormally dry. It is clear that for doughs which are neither exceptionally wet nor dry, there is a good general correlation, and this is especially noteworthy when one considers how very many assumptions are made in deriving equation (2).

Conclusions

The Chopin extensimeter, used in the usual manner in which all doughs are compared at the same moisture content, measures three factors:

(1) The maximum pressure attained when a bubble is blown in a strip of dough under standard conditions (P)
(2) The square root of the maximum volume (G)
(3) The area of the pressure-volume diagram (W). This is believed to depend partly on the property of the dough to allow gas to escape through it before final rupture and is therefore not considered in relation to viscosity and modulus measurements.

P is primarily related to the moisture absorbing capacity of the flour.

G is primarily related to a complex function of viscosity and modulus, but is approximately dependent on the product of a function known to be related to "spring" and one related to "shortness." Other factors undoubtedly enter to a lesser extent.

The baking quality of a flour depends primarily on spring and shortness, but it is not yet possible to say how far the composite function of them measured by the Chopin extensimeter corresponds with what is required for a good baking quality either under French or English conditions. This point requires further investigation.

Summary

A preliminary analysis of the physical properties of dough measured by the Chopin extensimeter indicates that water absorption capacity and a complex function of viscosity and modulus are the principal factors involved. Under the conditions of the test, the former is directly related to viscosity, and the latter depends on a complex mixture of "spring" and "shortness" which has been only partially resolved.

In view of the increasing use which is being made of the Chopin instrument as a criterion of wheat and flour quality independent of any baking test, the importance of a wider understanding of the nature of the factors measured is stressed.

In conclusion, one of the authors (G.W.S.B.) wishes to express his gratitude to M. Demolon, Inspecteur Général d'Agriculture de France, for the hospitality of the Centre National de Recherches Agronomiques de Versailles during the progress of this work, and to the Société pour la Vulgarisation des Engrais for the help which made possible his participation in it.

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A MOLDER FOR MICRO-BAKING

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(Received for publication August 10, 1936)

In 1932 Dr. E. E. Werner made a study (unpublished) of micro-baking procedures using 25 g. of flour. This modification of the standard test has been described by Geddes and Sibbitt [2] and Geddes and Aitken [3] who used the test in wheat varietal studies where only a small amount of flour was available.

The author, having had access to Werner's results, became interested in micro-baking as a possible better and more convenient means of making fermentation time-volume curves and other series bakes of similar nature. For this purpose the method apparently presented several desirable features chief of which was the ability to mix a number of loaves at one mixing in the ordinary small laboratory machine and thus eliminate the mixing variable in any one series.

Accordingly, many preliminary baking experiments were carried out using miniature tall-form aluminum pans and molding by hand and also with a rolling pin and rails of definite thicknesses. The small doughs proved somewhat difficult to pan and often gave unsymmetrical loaves. Results obtained, while satisfactory, indicated the need of mechanical aid in uniformly panning these small doughs. The apparatus described in the following paragraphs was designed and constructed for this purpose.

Description of Apparatus

The micro-molder consists of a sheeter unit and a compressor unit as shown in Figure 1. Side frames of the sheeter are cast iron. The sheeting rolls and shafts are integral turned from cold rolled steel and run in bronze bearings. The working surfaces of the rolls are 1.825 inches in diameter. Cut steel spur gears packed in grease in the gear case connect the two rolls. Side frames of the compressor are also cast iron with bronze bearings. The compression drum is a properly trued and faced cast wheel with a steel shaft. The drum is 8.0 inches in diameter with a 2.0 inch face. The compression board and side

[1] The author's thanks are due Dr. E. E. Werner for much helpful advice and collaboration.

[2] Geddes, W. F., and Sibbitt, L. D. Variability in experimental baking. IV. Studies on mixing, sheeting rolls, pan shape and 50 and 25 gram formulas. Cereal Chem. 10: 560–584 (1933).

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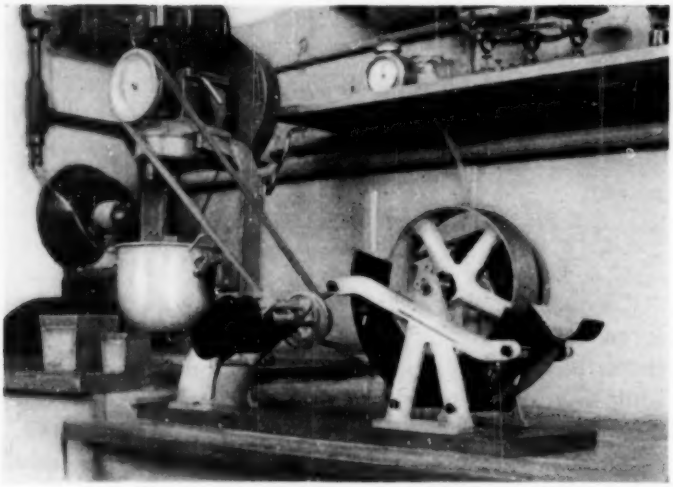

Figure 1. Sheeter and compressor units comprising micro-molder.

boards are 16 gauge steel. The side boards are permanently fixed in position and the compression board is adjustable and removable. A power take-off on the constant speed shaft of the Hobart mixer provides a convenient source of power without the use of any speed reduction device. V-belts are used on the drives. The sheeter rolls run 280 revolutions per minute and the compression drum runs 80 revolutions per minute. These speeds give the proper surface speeds for correct molding. The two units are mounted on a common base and the assembly weighs 28 pounds.

In operation the dough is passed through the sheeter, loosely curled up by hand and passed through the compressor unit from which it emerges perfectly rolled and sealed with the desired uniform appearance. Comparative sizes of regular and micro pans can also be seen in Figure 1.

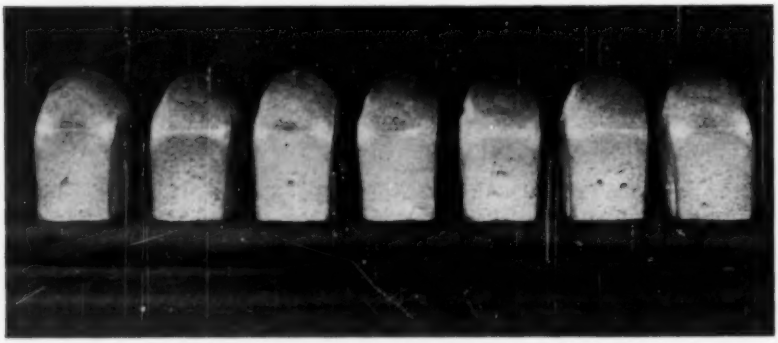

Figure 2. Replicate bakings illustrating type of loaf.

Results

For replication studies eight loaves were mixed and given the regular baking procedure. Seven loaves were baked and the eighth used to measure the volume of molded dough. Figure 2 shows a typical replicate bake. The volume spread between smallest and largest loaf in this particular test was 2 cc. In many other replication tests no volume spread of more than 5 cc. has been encountered. Figure 3 shows the inside appearance of the same loaves. Figure 4

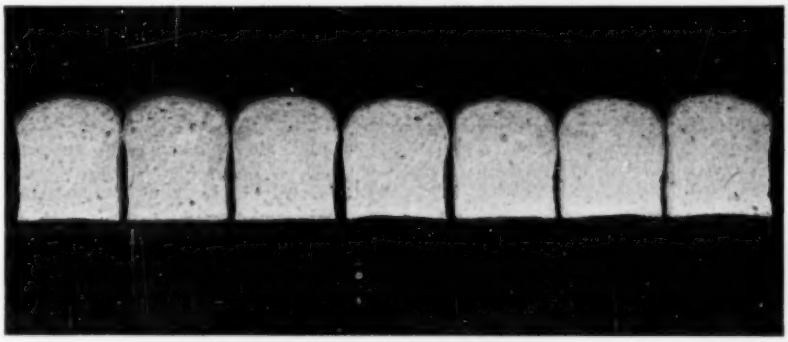

Figure 3. Inside appearance of micro loaves.

Figure 4. Micro loaves with no sugar in dough.

shows a characteristic series bake using no sugar in the dough and varying the fermentation time from one-half hour to five hours by one-half hour steps.

Use of the molder eliminated the lack of symmetry obtained in hand molding. The external and internal features of the micro loaves are fully comparable to those of the regular 100-g. loaves.

ERRATUM

STUDY OF SIZE OF BAKING PAN IN EXPERIMENTAL BAKING. By C. F. Davis. Volume XIV, pages 38, 39, 42, and 44. In Tables I, II, III, and V, last column reads "Average weight all doughs—Grams"; these column headings should read "Average, all dough weights."

BOOK REVIEW

Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists. Fourth Edition, 1935. 710 pages, 52 illustrations, and 24 reference tables. Published by the A. O. A. C., Box 540 Benjamin Franklin Station, Washington, D. C. Price $5.00 ($4.00 to members when ordered through the office of the Secretary-Treasurer. Special discounts also are given when ordering 5 or more copies.

The fourth edition of the Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists is now ready for distribution. It represents a vast amount of careful work on the part of the committee on editing methods of analysis since the publication of the third edition in 1930.

Although the text follows the same general style of presentation characteristic of previous editions there has been a regrouping of methods in several instances according to subject matter which is a decided improvement. Thus, Chapter XVII of the third edition, relative to beers, wines, and distilled liquors, has been rewritten and enlarged and now forms the subject matter of Chapters XIV, XV, and XVI on malt, beverages, wines, and distilled liquors.

Many of the methods described in the third edition have been rewritten in the interest of clarity and easy reading.

The fourth edition is an enlargement over the last edition as it contains over 125 additional pages. This increase in size is not due solely to added chapters on special subjects, but is occasioned by the inclusion of newer and more complete methods in the individual chapters listed in the book.

An added feature is a new alcohol table. Refinements have been made in the refractometric tables for sucrose determinations.

The section on cereal foods has been enlarged 30 percent by the inclusion of several new methods such as the Gustafson method for determining the original ash content of phosphated and self-rising flours, methods for determining the benzoyl peroxide bleach in flour, detection of rye flour in wheat flour, diastatic activity of flour, viscosity of flour-water suspensions, and methods for the analysis of bread and for baked products other than bread (alimentary pastes).

For the use of the cereal chemist, there is included, on a tentative basis, the methods of analysis for malt adopted by the American Society of Brewing Chemists. Methods are also described for the analysis of the more prominent malt adjuncts.

In appearance the book is more attractively bound than any of the previous editions. This, in consideration of the vast storehouse of information contained, is an added reason why the fourth edition of the Methods of Analysis of the A. O. A. C. should find a place on the book-shelf of every cereal chemist.

D. A. COLEMAN